PCT

NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

CIBA SPECIALTY CHEMICALS HOLDING INC.
Patentabteilung Ressort P/TM/SI LE 5
Klybeckstrasse 141
CH-4057 Basel SUISSE
PATA RATH SES
HPF

2 2

IMPORTANT NOTICE

From the INTERNATIONAL BUREAU

Date of mailing (day/month/year)

28 December 2000 (28.12.00)

Applicant's or agent's file reference

International application No. PCT/EP00/05314

HP/2-22037/A

International filing date (day/month/year)

08 June 2000 (08.06.00)

Priority date (day/mont//year)
18 June 1999 (18.06.99)

Applicant

CIBA SPECIALTY CHEMICALS HOLDING INC. et al

 Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice: AG,AU,DZ,KP,KR,MZ,US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AE,AL,AM,AP,AT,AZ,BA,BB,BG,BR,BY,CA,CH,CN,CR,CU,CZ,DE,DK,DM,EA,EE,EP,ES,FI,GB,GD,GE,GH,GM,HR,HU,ID,IL,IN,IS,JP,KE,KG,KZ,LC,LK,LR,LS,LT,LU,LV,MA,MD,MG,MK,MN,MW,MX,NO,NZ,OA,PL,PT,RO,RU,SD,SE,SG,SI,SK,SL,TJ,TM,TR,TT,TZ,UA,UG,UZ,VN,YU,ZA,ZW The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on 28 December 2000 (28.12.00) under No. WO 00/78277

REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the national phase, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

The International Bur au f WIPO 34, ch min d s C I mbett s 1211 Gen va 20, Switzerland Authorized officer

J. Zahra

Facsimile No. (41-22) 740.14.35

Telephone No. (41-22)/389-83.38

3735985

Form PCT/IB/308 (July 1996)

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REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

PCT/EP 00 / 05314

International Application No.

(08.06.2000)

08 JUNE 2000

International Filing Date

EUROPEAN PATENT OFFICE PCT INTERNATIONAL APPLICATION

Name of receiving Office and "PCT International Application"

	Applicant's or agent's fi			-22037/A	
Box No. I TITLE OF INVENTION			···· <u>···</u> ·/		
Micropigment mixture					
Box No. II APPLICANT		-	. 44 .		
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.). Ciba Specialty Chemicals Holding Inc.				person is also inventor	
Klybeckstrasse 141			Telephone No. +41 61 636 11 11		
4057 Basel CH		ŀ	Facsimile No. +41 61 636 79 76		
		}	Teleprinter No.	41 01 000 13 70	
State (that is, country) of nationality: CH	State (that is, country) of r	esidence:	СН		
	stated States except States of America	L L	United States merica only	the States indicated in the Supplemental Box	
Box No. III FURTHER APPLICANT(S) AND/OR (
Name and address: (Family name followed by given name; for a legal entity must include postal code and name of country. The country of the address in State (that is, country) of residence if no State of residence is indicated below.	ndicated in this Rox is the applica-	dress ant's	This person is	s:	
LUTHER, Helmut Tüllingerweg 3a 79639 Grenzach-Wyhlen DE applicant only applicant and inventor inventor only (If this check marked, do not fill in below		cant and inventor			
State (that is, country) of nationality: DE	State (that is, country) of r	esidence:	DE		
	ated States except States of America	<u> </u>	United States Imerica only	the States indicated in the Supplemental Box	
Further applicants and/or (further) inventors are in	dicated on a continuation	sheet.			
Box No. IV AGENT OR COMMON REPRESENTA	TIVE; OR ADDRESS	FOR CO	DRRESPOND	ENCE	
The person identified below is hereby/has been appointed to act of the applicant(s) before the competent International Authorities	on behalf as:	igent	[3	common representative	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) Telephone No. +41 61 636 11 11					
Ciba Specialty Chemicals Holding Inc. Patent Department Klybeckstrasse 141 Facsimile No. +41 61 636 79 76			+41 61 636 79 76		
4057 Basel CH Teleprinter No.			,		
Address for correspondence: Mark this check-ba	ox where no agent or com	mon rep	resentative is/1	nas been appointed and the	
space above is used instead to indicate a special address to which correspondence should be sent.					

Box N	lo V	DESIGNATION OF STATES			
		DESIGNATION OF STATES		_	
	he following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):				
Region	nal Paten	t ·			
X	AP	ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesoti Republic of Tanzania, UG Uganda, ZW Zimbabwe, and any oth	ho, MW er State	/ Malawi, which is	SD Sudan, SL Sierra Leone, SZ Swaziland, TZ United a Contracting State of the Harare Protocol and of the PCT
X	EA	Republic of Tanzania, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT			
M	EP	European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT			
X	OA	Portugal. SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment desired, specify on dotted line)			
Nation	al Pateni	(if other kind of protection or treatment desired, specify on dotte			
\mathbf{x}	AE	United Arab Emirates	u une):	LR	Liboria
\boxtimes	AL	Albania	X	LS	Liberia
X	AM	Armenia	X	LT	
X	AT	Austria	×		Lithuania
$\overline{\mathbf{Z}}$	AU		<u> </u>	LU	Luxembourg
X		Australia		LV	Latvia
\mathbf{x}	AZ	Azerbaijan	X	MA	Morocco
	BA	Bosnia and Herzegovina	X	MD	Republic of Moldova
X	BB	Barbados	X	MG	Madagascar
\square	BG	Bulgaria	\mathbf{X}	MK	The former Yugoslav Republic of Macedonia
\boxtimes	BR	Brazil			
\boxtimes	BY	Belarus	\mathbf{X}	MN	Mongolia
X	CA	Canada	\boxtimes	MW	Malawi
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\square	CN	China	\mathbf{X}	NO	Norway
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X	CU	Cuba	\boxtimes	PL	
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X	DE	Germany	X	PT	Portugal
$\overline{\boxtimes}$	DK	Denmark	X	RO	Romania
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X		Dominica		SD	Sudan
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X	ES	Spain	X	SG	Singapore
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X	GB	United Kingdom	X	SK	Slovakia
Σ.	GD	Grenada	\square	SL	Sierra Leone
X	GE	Georgia	\mathbf{X}	TJ	Tajikistan
	GH	Ghana	\square	TM	Turkmenistan
X	GM	Gambia	\boxtimes	TR	Turkey
\boxtimes	HR	Croatia	\boxtimes	TT	Trinidad and Tobago
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\boxtimes	ID	Indonesia	X	UA	Ukraine
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[X]	IN	India	\boxtimes	US	United States of America
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Ø	V D	Danible of Vo.	⊠	zw	Zimbabwe
	KR K7	Republic of Korea			eserved for designating States (for the purposes of a national
	KZ	Kazakstan			have become party to the PCT after issuance of this sheet:
į Σ	LC	Saint Lucia	X	DZ	Algeria
123	LK	Sri Lanka	X	AG	Antigua and Barbuda
			\boxtimes	MZ	Mozambique

Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.)

Sheet 3

Box No. VI PRIORITY CLAIM			·	
- Thirding Commit		Further priori		in the Supplemental Box
Filing Date of earlier application (day/month/year)	Number of earlier application	national application:	Where earlier application regional application:	international application:
item (1) 18th June 1999 (18.06.99)	99810543.1	country	regional Office	receiving Office
item (2)				
item (3)				
The receiving Office is requested to preport of the earlier application(s) (only if the of the present international application * Where the earlier application is an ARIPO a	carlier application was filed is the receiving Office) ideo oplication, it is mandatory to	with the Office which for the trified above as item(s):	the purposes	try party to the Paris
Convention for the Protection of Industrial Pro- Box No. VII INTERNATIONAL SEA			e 4.10(b)(ii). See Supplem	ental Box.
Choice of International Searching Authority (If two or more International Searching Authorities are competent to carry out the international search, indica Authority chosen; the two-letter code may be used):	(ISA) Request to use	results of earlier search; equested from the International	d Searching Authority):	(if an earlier search has been ntry (or regional Office)
ISA/	02/03/00	99 81		EP
Box No. VIII CHECK LIST; LANGUA				
This international application contains the following number of sheets:	This international applica	ition is accompanied by	the item(s) marked below:	
request : 4	1. A fee calculation	n sheet		
description (excluding : 49 sequence listing part)	J [ed power of attorney		
claims : 11		ral power of attorney; refer	rence number, if any:	
abstract : 1		plaining lack of signature ment(s) identified in Box N	No VI as item(s): (1)	
drawings :		international application is	• •	
sequence listing part of description : —		ations concerning deposite	ed microorganism or other	biological material
Total number of sheets : 65	8. nucleotide and 9. other (specify	d/or amino acid sequence l):	listing in computer readab	le form
Figure of the drawings which should accompany the abstract:		Language of filing of international applicatio	the German	
Box No. IX SIGNATURE OF APPLI				
Next to each signature, indicate the name of the he request)	person signing and the cape	acity in which the person s	igns (if such capacity is no	ot obvious from reading
		Ciba Specia	Ity Chemicals Hol	ding Inc.
		i.V.	O. Spengles	-
07.06.2000		_	erena Spendler stent Administrator	
	For receiving C	Office use only		
Date of actual receipt of the purported international application:	(08.06.	•	000	2. Drawings:
 Corrected date of actual receipt due to later timely received papers or drawings complet the purported international application: 	out ing			not
Date of timely receipt of the required corrections under PCT Article 11(2):				received:
International Searching Authority specified by the applicant:	-	6. Transmittal of until search for	search copy delayed	
	For International	Bureau use only		
Date of receipt of the record copy		•,		

Form PCT/RO/101 (last sheet) (January 2000)

See Notes to the request form

VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS

PCT

REC'D 1 6 JUL 2001
WIPO PCT

INTERNATIONALER VORLÄUFIGER PRÜFUNGSBERICHT

(Artikel 36 und Regel 70 PCT)

Aktenzeic	nen de	es Anmelders oder Anwalts			
HP/2-22			WEITERES VOR	siehe Mittei Vorläufigen	lung über die Übersendung des internationalen Prüfungsberichts (Formblatt PCT/IPEA/416)
Internation	ales A	Aktenzeichen	Internationales Anmeld	edatum <i>(Tag/Monat/Jahr)</i>	Prioritätsdatum (Tag/Monat/Tag)
PCT/EP	00/05	5314	08/06/2000		18/06/1999
Internation A61K7/4		atentklassifikation (IPK) oder r	nationale Klassifikation ur	nd IPK	
Anmelder	,		· · · · · · · · · · · · · · · · · · ·		
CIBA SF	PECIA	ALTY CHEMICALS HOL	LDING INC. et al.		-
1. Diese Behö	er inte rde e	ernationale vorläufige Prüf rstellt und wird dem Anme	ungsbericht wurde vo elder gemäß Artikel 36	n der mit der internatio übermittelt.	nalen vorläufigen Prüfung beauftragten
2. Diese	er BEI	RICHT umfaßt insgesamt	5 Blätter einschließlich	ch dieses Deckblatts.	
u	nd/oc	der Zeichnungen, die geär	ndert wurden und dies	em Bericht zugrunde I	ter mit Beschreibungen, Ansprüchen iegen, und/oder Blätter mit vor dieser t 607 der Verwaltungsrichtlinien zum PCT).
Diese	Anla	gen umfassen insgesamt	Blätter.		
3. Diese	r Beri	icht enthält Angaben zu fo	olgenden Punkten:		
1	\boxtimes	Grundlage des Berichts			
11		Priorität			
Ш		Keine Erstellung eines G	autachtens über Neuh	eit, erfinderische Tätig	keit und gewerbliche Anwendbarkeit
IV		MangeInde Einheitlichke		-	
V	×	Begründete Feststellung gewerblichen Anwendba	nach Artikel 35(2) hin rkeit; Unterlagen und	sichtlich der Neuheit, Erklärungen zur Stütz	der erfinderischen Tätigkeit und der ung dieser Feststellung
VI		Bestimmte angeführte U		-	
VII		Bestimmte Mängel der in	nternationalen Anmeld	ung	
VIII		Bestimmte Bemerkunger	n zur internationalen A	nmeldung	
Datum der l	Einreic	hung des Antrags		Datum der Fertigstellun	g dieses Berichts
23/11/200	00			12.07.2001	
		schrift der mit der internationa ten Behörde:	alen vorläufigen	Bevollmächtigter Bedier	nsteter September AND THE PROPERTY AND T

Ortega Plaza, M.D.

Tel. Nr. +49 89 2399 8284

Europäisches Patentamt D-80298 München

Tel. +49 89 2399 - 0 Tx: 523656 epmu d

INTERNATIONALER VORLÄUFIGER PRÜFUNGSBERICHT

Internationales Aktenzeichen PCT/EP00/05314

l.	Grundlag	des	Berichts
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1.	Au eir	ıfforderung nach Arti	ndteile der internationalen Anmeldung (<i>Ersatzblätter, die dem Anmeldeamt auf eine</i> kel 14 hin vorgelegt wurden, gelten im Rahmen dieses Berichts als "ursprünglich nm nicht beigefügt, weil sie keine Änderungen enthalten (Regeln 70.16 und 70.17)): :
	1-4	19	ursprüngliche Fassung
	Pa	tentansprüche, Nr.:	
	1-3	31	ursprüngliche Fassung
2.	die unt Die	internationale Anme er diesem Punkt nicl Bestandteile stande	e: Alle vorstehend genannten Bestandteile standen der Behörde in der Sprache, in der eldung eingereicht worden ist, zur Verfügung oder wurden in dieser eingereicht, sofern anderes angegeben ist.
	eini	gereicht; dabei hand die Sprache der Üb Regel 23.1(b)).	eit es sich um bersetzung, die für die Zwecke der internationalen Recherche eingereicht worden ist (nac
		die Veröffentlichung	gssprache der internationalen Anmeldung (nach Regel 48.3(b)).
			ersetzung, die für die Zwecke der internationalen vorläufigen Prüfung eingereicht worder
3.	Hin: inte	sichtlich der in der in rnationale vorläufige	ternationalen Anmeldung offenbarten Nucleotid- und/oder Aminosäuresequenz ist die Prüfung auf der Grundlage des Sequenzprotokolls durchgeführt worden, das:
		in der internationale	en Anmeldung in schriftlicher Form enthalten ist.
			internationalen Anmeldung in computerlesbarer Form eingereicht worden ist.
			chträglich in schriftlicher Form eingereicht worden ist.
			chträglich in computerlesbarer Form eingereicht worden ist.
		Die Erklärung, daß	das nachträglich eingereichte schriftliche Sequenzprotokoll nicht über den ter internationalen Anmeldung im Anmeldezeitpunkt hinausgeht, wurde vorgelegt.
		Die Erklärung, daß Sequenzprotokoll e	die in computerlesbarer Form erfassten Informationen dem schriftlichen ntsprechen, wurde vorgelegt.
Į.	Aufg	grund der Änderunge	en sind folgende Unterlagen fortgefallen:
		Beschreibung,	Seiten:
		Ansprüche,	Nr.:
		Zeichnungen,	Blatt:
		-	

INTERNATIONALER VORLÄUFIGER PRÜFUNGSBERICHT

Internationales Aktenzeichen PCT/EP00/05314

5. Dieser Bericht ist ohne Berücksichtigung (von einigen) der Änderungen erstellt worden, da diese aus den angegebenen Gründen nach Auffassung der Behörde über den Offenbarungsgehalt in der ursprünglich eingereichten Fassung hinausgehen (Regel 70.2(c)).

(Auf Ersatzblätter, die solche Änderungen enthalten, ist unter Punkt 1 hinzuweisen;sie sind diesem Bericht beizufügen).

1-31

- 6. Etwaige zusätzliche Bemerkungen:
- V. Begründete Feststellung nach Artikel 35(2) hinsichtlich der Neuheit, der erfinderischen Tätigkeit und dir gewerblichen Anwendbarkeit; Unterlagen und Erklärungen zur Stützung dieser Feststellung
- 1. Feststellung

Neuheit (N) Ja: Ansprüche

Nein: Ansprüche 1-31

Erfinderische Tätigkeit (ET)

Ja: Ansprüche Nein: Ansprüche

Gewerbliche Anwendbarkeit (GA) Ja: Ansprüche 1-31

Nein: Ansprüche

2. Unterlagen und Erklärungen siehe Beiblatt

Zu Punkt V

Begründete Feststellung nach Regel 66.2(a)(ii) hinsichtlich der Neuheit, der erfinderischen Tätigkeit und der gewerblichen Anwendbarkeit; Unterlagen und Erklärungen zur Stützung dieser Feststellung

Folgende Dokumenten werden für die Erstellung des vorliegenden vorläufigen 1. Berichts in Betracht gezogen:

D1 = US-A-5445815

D2 = EP-A-0821939

D3 = WO-A-9700851 (in der Beschreibung erwähnt)

D4 = US-A-5518713 (in der Beschreibung erwähnt)

D5 = US-A-5338539 (in der Beschreibung erwähnt)

D6 = EP-A-0582189 (in der Beschreibung erwähnt)

D7 = EP-A-0818450 (in der Beschreibung erwähnt)

D8 = EP-A-0654469 (in der Beschreibung erwähnt)

D9 = US-A-5601811 (in der Beschreibung erwähnt)

D10 = Dr. U. Schöffling, Trier, Arzneiformenlehre, DAV, Stuttgart 1998

D11 = Pflegekosmetik, W. Raab, U. Kindl, Wissenschaftliche Verlagsgesellschaft mbH Stuttgart, 1999.

2. Anspruch 1 bezieht sich auf die Verwendung von Mischungen aus mikronisierten organischen UV-Filtern zum Schützen der menschlichen und tierischen Haut und Haare vor der schädigenden Einwirkung von UV-Strahlung.

Die Verwendung von organischen UV-Filtern zum Schützen der menschlichen Haut und Haare vor der schädlichen Einwirkung von UV-Strahlung ist alt bekannt (siehe u.a. D1-D9). Die Verwendung von Mischungen aus organischen UV-Filtern, damit man ein breiteres UV-Spektrum deckt (siehe u.a. D11) ist auch allkömmlich. Ferner bleibt im Anspruch 1 nicht definiert um welche "Mischungen" es sich handelt. Die Verwendung in Anspruch 1 der Ausdruck "mikronisierten" reicht nicht um eindeutigt Neuheit gegenüber u.a. D1 herzustellen. "Mikronisierten" ist vaage und solange keine genaue Grösseangabe angegeben wird, wird als Synonim für "feinteilig" angesehen. Ferner, ist die Verwendung von feinteiligen und mikronisierten Produkten eine übliche Technologie in der Bereich von

Arzneiformlehre (siehe u.a. D10).

D1 beschreibt "Composite", erhältlich durch Zusammenschmelzen von mindestens zwei organischen UV-Filtern. Daher ist der Gegenstand von Anspruch 18 auch nicht neu.

Die in den Ansprüche 29 und 31 beanspruchten kosmetischen oder pharmazeutischen Formulierungen umfassen alle im Stand der Technik beschriebenen Formulierungen, die organischen Filtern beinhalten (siehe D1-D9), da es aus dem Wortlaut dieser Ansprüche nicht eindeutigt herausgeht, ob die UV-Filtern in mikronisierten Form vorliegen und um welche Mischungen tatsächlich es sich handelt (aus der gleichen oder unterschiedlichen Verbindungsklasse).

Die obige Analyse gilt sinngemäß für den Gegenstand allen anderen Ansprüche. Weitere Merkmale entsprechen allkömmlichen Merkamle aus der Gebiet von Zubereitungen mit UV-Filtern (siehe u.a. D11).

Daher ist es z.Z. nicht offensichtlich worin eine Erfindung liegt für die vorliegende Anmeldung.



PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference HP/2-22037/PCT/A	FOR FURTHER ACTION	Examination	ionofTransmittalofInternational Preliminary Report (Form PCT/IPEA/416)		
International application No. PCT/EP00/05314	International filing date (day/ 08 June 2000 (08.0		Priority date (day/month/year) 18 June 1999 (18.06.99)		
International Patent Classification (IPC) or national classification and IPC A61K 7/42					
Applicant CIBA S	SPECIALTY CHEMICA	LS HOLDIN	NG INC.		
and is transmitted to the applicant a	 This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36. 				
2. This REPORT consists of a total of	5 sheets, includ	ling this cover	sheet.		
This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).			ion, claims and/or drawings which have been ations made before this Authority (see Rule		
These annexes consist of a to	otal of sheets.				
This report contains indications relations	ating to the following items:				
I Basis of the report					
II Priority	Priority				
III Non-establishment	of opinion with regard to nove	elty, inventive s	step and industrial applicability		
IV Lack of unity of in	vention				
V Reasoned statemen	nt under Article 35(2) with regainstions supporting such statem	ard to novelty, i nent	inventive step or industrial applicability;		
VI Certain documents	cited				
VII . Certain defects in	the international application				
VIII Certain observatio	ns on the international applicat	tion			
Date of submission of the demand	Date	e of completion	of this report		
23 November 2000 (2			2 July 2001 (12.07.2001)		
Name and mailing address of the IPEA/El	P Aut	thorized officer			
Facsimile No.	Tel	ephone No.			

Translation



INTERNATIONAL PRELIMINARY EXAMINATION REPORT

- 45020			
		application	No.

PCT/EP00/05314

1. With regard to the elements of the international application:*	<u> </u>
	i
the international application as originally filed	
the description:	s originally filed
pages 1-49 , a , filed	
pages, mee	
pages, filed with the letter of	
the claims:	
pages 1-31 , a	is originally filed
as amended (together with any statement	with the demand
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nages , Illed	with the demand
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 With regard to the language, all the elements marked above were available or furnished to this Authority in the the international application was filed, unless otherwise indicated under this item. These elements were available or furnished to this Authority in the following language the language of a translation furnished for the purposes of international search (under Rule 23.1(b)). the language of publication of the international application (under Rule 48.3(b)). the language of the translation furnished for the purposes of international preliminary examination (under 55.3). With regard to any nucleotide and/or amino acid sequence disclosed in the international application, preliminary examination was carried out on the basis of the sequence listing:	der Rule 55.2 and/ the international
4. The amendments have resulted in the cancellation of: the description, pages the claims, Nos. the drawings, sheets/fig This report has been established as if (some of) the amendments had not been made, since they have be beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).** * Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article in this report as "originally filed" and are not annexed to this report since they do not contain amend and 70.17). ** Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report	le 14 are referred to dments (Rule 70.16

INTERNATIONAL PRESENTARY EXAMINATION REPORT

V. Reasoned statement under Article citations and explanations supporti	35(2) with regard to novelty ng such statement	, inventive step or industrial app	licability;
Statement			
Novelty (N)	Claims		YES
	Claims	1-31	NO
Inventive step (IS)	Claims		YES
	Claims	1-31	NO
Industrial applicability (IA)	Claims	1-31	YES
	Claims		NO

- Citations and explanations
 - 1. In establishing the present preliminary report, reference is made to the following documents:

D1: US-A-5 445 815

D2: EP-A-0 821 939

D3: WO-A-97/00851 (cited in the description)

D4: US-A-5 518 713 (cited in the description)

D5: US-A-5 338 539 (cited in the description)

D6: EP-A-0 582 189 (cited in the description)

D7: EP-A-0 818 450 (cited in the description)

D8: EP-A-0 654 469 (cited in the description)

D9: US-A-5 601 811 (cited in the description)

D10: SCHÖFFLING DR U, Trier, ARZNEIFORMENLEHRE,

DAV, Stuttgart, 1998

D11: RAAB W AND KINDL U, PFLEGEKOSMETIK,

WISSENSCHAFTLICHE VERLAGSGESELLSCHAFT MBH,

Stuttgart, 1999.

2. Claim 1 relates to the use of mixtures of micronised organic UV filters to protect human and animal skin and hair against the harmful effect of UV radiation.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

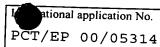
The use of organic UV filters to protect human and animal skin and hair against the harmful effect of UV radiation has long been known (see inter alia D1 to D9). The use of mixtures of organic UV filters, in order to cover a broader UV spectrum (see inter alia D11), is also generally known. Moreover it remains undefined in Claim 1 what "mixtures" are of interest. The use in Claim 1 of the term "micronised" is not adequate to establish clearly novelty over, inter alia, D1. "Micronised" is vague and in the absence of precise dimensional information is taken to be a synonym for "finely divided". Moreover the use of finely divided and micronised products is conventional in the field of pharmacology – see inter alia D10.

D1 describes "composites" obtained by combining at least two organic UV filters. Therefore the subject matter of Claim 18 is not novel.

The cosmetic or pharmaceutical formulations claimed in Claims 29 and 31 encompass all formulations with organic filters described in the prior art (see D1 to D9), since it is not clear from the wording of said claims whether micronised UV filters are present nor exactly what mixtures (of identical or different classes of compound) are of interest.

The above analysis clearly applies likewise to the subject matter of all the other claims. Other features correspond to conventional features from the field of UV filter preparations - see *inter alia* D11.

/...



	Therefore, at present it is not evident what
•	invention is claimed in the present application.
	- · · · · · · · · · · · · · · · · · · ·
•	

PCT

INTERNATIONALER RECHERCHENBERICHT

(Artikel 18 sowie Regeln 43 und 44 PCT)

Aktenzeichen des Anmelders oder Anwalts HP/2-22037/A	WEITERES VORGEHEN	Recherchenberichts (F	iehe Mitteilung über die Übermittlung des internationalen Recherchenberichts (Formblatt PCT/ISA/220) sowie, soweit utreffend, nachstehender Punkt 5				
Internationales Aktenzeichen	Internationales Anmeldedatum (Tag/Monat/Jahr)		(Frühestes) Prioritätsdatum (Tag/Monat/Jahr)				
PCT/EP 00/05314	08/06/2	000	18/06/1999				
CIBA SPECIALTY CHEMICALS HO	OLDING INC.						
Dieser internationale Recherchenbericht wurd Artikel 18 übermittelt. Eine Kopie wird dem Int Dieser internationale Recherchenbericht umfa	ternationalen Büro übern		rstellt und wird dem Anmelder gemäß				
			Unterlagen zum Stand der Technik bei.				
Grundlage des Berlchts							
 a. Hinsichtlich der Sprache ist die inter durchgeführt worden, in der sie eing 	rnationale Recherche au pereicht wurde, sofern un	f der Grundlage der inter ter diesem Punkt nichts a	rnationalen Anmeldung in der Sprache anderes angegeben ist.				
Die internationale Recherch Anmeldung (Regel 23.1 b))	e ist auf der Grundlage e durchgeführt worden.	einer bei der Behörde ein	gereichten Übersetzung der internationalen				
b. Hinsichtlich der in der internationale Recherche auf der Grundlage des S in der internationalen Anmel	Gequenzprotokolls durcho	geführt worden, das	Aminosäuresequenz ist die internationale				
zusammen mit der internatio	onalen Anmeldung in cor	nputerlesbarer Form eing	gereicht worden ist.				
bei der Behörde nachträglich	h in schriftlicher Form eir	ngereicht worden ist.					
bei der Behörde nachträglich	h in computerlesbarer Fo	orm eingereicht worden is	st.				
Die Erklärung, daß das nach internationalen Anmeldung i			oll nicht über den Offenbarungsgehalt der t.				
Die Erklärung, daß die in ∞ wurde vorgelegt.	mputerlesbarer Form erf	aßten Informationen dem	n schriftlichen Sequenzprotokoll entsprechen,				
2. Bestimmte Ansprüche hab	en sich als nicht reche	e rchlerbar erwlesen (sie	ehe Feld I).				
3. Mangelnde Einheitlichkeit	der Erfindung (siehe Fe	eld II).					
4. Hinsichtlich der Bezeichnung der Erfin	dung						
xird der vom Anmelder eing	wird der vom Anmelder eingereichte Wortlaut genehmigt.						
wurde der Wortlaut von der l	Behörde wie folgt festge:	setzt:					
5. Hinsichtlich der Zusammenfassung							
	gel 38.2b) in der in Feld innerhalb eines Monats	III angegebenen Fassun	g von der Behörde festgesetzt. Der osendung dieses internationalen				
6. Folgende Abbildung der Zelchnungen is	st mit der Zusammenfass	sung zu veröffentlichen: /	Abb. Nr.				
wie vom Anmelder vorgesch	lagen		keine der Abb.				
weil der Anmelder selbst kei	• •	~					
weil diese Abbildung die Erfi	ndung besser kennzeich	net.					

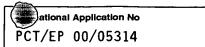
INTERNATIONALER SECHERCHENBERICHT

In conales Aktenzeichen PCT/EP 00/05314

A. KLASS IPK 7	ifizierung des anmeldungsgegenstandes A61K7/42		
Nach der In	nternationalen Patentklassifikation (IPK) oder nach der nationalen Kla	assifikation und der IPK	
	RCHIERTE GEBIETE erter Mindestprüfstoff (Klassifikationssystem und Klassifikationssymb	-1-1	
IPK 7	A61K	ю е ј	
Recherchie	orte aber nicht zum Mindestprüfstoff gehörende Veröffentlichungen, s	oweit diese unter die recherchierten Gebiete	e fallen
1	er internationalen Recherche konsultierte elektronische Datenbank (f	Name der Datenbank und evtl. verwendete	Suchbegriffe)
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Nategone	Bezeichnung der Veronentlichlung, soweit enfolderlich sinter 20.322	O GELIU PELISCUI KOMIHENORI 1 ene	Betr. Anspruch Nr.
х	US 5 445 815 A (R. SIEGFRIED) 29. August 1995 (1995-08-29) das ganze Dokument		1,18
А	EP 0 821 939 A (3V SIGMA S.P.A.) 4. Februar 1998 (1998-02-04) Beispiel 2		1
entre	tere Veröffentlichungen sind der Fortsetzung von Feld C zu lehmen	χ Siehe Anhang Patentfamilie	
"A" Veröffer aber ni "E" älteres l Anmel "L" Veröffer schein andere soll od ausgef "O" Veröffer eine B "P" Veröffer dem be	entlichung, die sich auf eine mündliche Offenbarung, lenutzung, eine Ausstellung oder andere Maßnahmen bezieht ntlichung, die vor dem internationalen Anmeldedatum, aber nach eanspruchten Prioritätsdatum veröffentlicht worden ist	 "T" Spätere Veröffentlichung, die nach dem oder dem Prioritätsdatum veröffentlicht Anmeldung nicht kollidiert, sondem nu Erfindung zugrundeliegenden Prinzips Theorie angegeben ist "X" Veröffentlichung von besonderer Bedekann allein aufgrund dieser Veröffentlicher in dieser Veröffentlicher Tätigkeit beruhend betra "Y" Veröffentlichung von besonderer Bedekann nicht als auf erfinderischer Tätigk werden, wenn die Veröffentlichung mit Veröffentlichungen dieser Kategorie in diese Verbindung für einen Fachmann "&" Veröffentlichung, die Mitglied derselben 	t worden ist und mit der rr zum Verständnis des der oder der ihr zugrundeliegenden utung; die beanspruchte Erfindung chung nicht als neu oder auf achtet werden utung; die beanspruchte Erfindung weit berühend betrachtet einer oder mehreren anderen Verbindung gebracht wird und naheliegend ist
Datum des /	Abschlusses der internationalen Recherche	Absendedatum des internationalen Re	cherchenberichts
1:	3. Oktober 2000	20/10/2000	
Name und P	Postanschrift der Internationalen Recherchenbehörde Europäisches Patentamt, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016	Bevollmächtigter Bediensteter Glikman, J-F	

INTERNATIONAL SEARCH REPORT

ation on patent family members



Patent document cited in search report		Publication date	Patent family member(s)		Publication date	
,	US. 5445815	Α	29-08-1995	US	5676934 A	14-10-1997
	EP 821939	Α	04-02-1998	NONE		

Us of mixtures of micropigments for preventing tanning and for lightening skin and hair

The present invention relates to the use of mixtures of micronized organic UV filters for preventing tanning and for lightening human skin and hair and to their use in cosmetic and pharmaceutical formulations.

It is known that certain organic UV filters, for example sparingly soluble benzotriazole or triazine compounds, have excellent UV filter properties if they are in micronized form.

Particularly in Asiatic countries, there is great interest in light protection filters or mixtures of light protection filters which preserve the colour of the skin following solar irradiation and, moreover, are able to impart a lighter appearance to the skin.

The object of the present invention is therefore to find micronized organic UV filters which prevent tanning of the skin and at the same time are able to lighten the skin.

Surprisingly, we have now found that micronized organic UV filters or mixtures of at least two micronized UV filters can achieve this object.

The present invention therefore provides for the use of mixtures of micronized organic UV filters for preventing tanning and for lightening of human skin.

Suitable UV filters which can be used according to the invention are organic, sometimes sparingly soluble, compounds, for example triazine derivatives, in particular hydroxyphenyltriazine compounds or benzotriazole derivatives, amides containing a vinyl group, cinnamic acid derivatives, sulfonated benzimidazoles, Fischer base derivatives, diphenylmalonitriles, oxalylamides, camphor derivatives, diphenylacrylates, paraaminobenzoic acid (PABA) and derivatives thereof, salicylates, benzophenones and also other classes of substance known as UV filters.

Preferred triazine derivatives which can be used according to the invention correspond to the formula

in which

 R_1 , R_2 and R_3 , independently of one another, are hydrogen; OH; C_1 - C_{18} alkoxy; -NH₂; -NH-R₄; -N(R₄)₂; -OR₄,

R₄ is C₁-C₅alkyl; phenyl, phenoxy, anilino or pyrrolo which are unsubstituted or substituted by one, two or three OH groups, carboxyl, -CO-NH₂, C₁-C₅alkyl or C₁-C₅alkoxy; a methylidenecamphor group; a group of the formula

corresponding alkali metal, ammonium, mono-, di- or tri- C_1 - C_4 alkylammonium, mono-, di- or tri- C_2 - C_4 alkanolammonium salts, or C_1 - C_3 alkyl esters thereof; or a radical of the

formula (1a)
$$-(CH_2)_{m_1} \stackrel{O}{\underset{R_5}{\downarrow}}$$
;

R₅ is hydrogen; unsubstituted C₁-C₅alkyl or C₁-C₅alkyl substituted by one or more OH groups; C₁-C₅alkoxy; amino; mono- or di-C₁-C₅alkylamino; M; a radical of the formula

(1b)
$$\begin{array}{c} HO \\ OH \\ OH \\ NH \end{array}$$
 OH; (1c) $R^{n} - \stackrel{N}{N} - (CH_{2})_{m_{3}} O - ; (1d) \qquad R^{n} - \stackrel{R'}{N} O^{-} ; or$

(1e)
$$-N \longrightarrow_{CO_2R_8}$$
; in which

R', R" and R", independently of one another, are unsubstituted C₁-C₁₄alkyl or C₁-C₁₄alkyl substituted by one or more OH groups;

 R_6 is hydrogen; M; C_1 - C_5 alkyl; or a radical of the formula $-(CH_2)_{m_2}$ -O- T_1 ;

M is a metal cation;

T₁ is hydrogen; or C₁-C₈alkyl;

m is 0 or 1

m₂ is 1 to 4; and

Further preferred triazine derivatives which can be used according to the invention correspond to the formula

in which

R₇ and R₈, independently of one another, are C₁-C₁₈alkyl; C₂-C₁₈alkenyl; a radical of the formula -CH₂-CH(-OH)-CH₂-O-T₁; or

 R_7 and R_8 are a radical of the formula (2a) $R_9 = \begin{bmatrix} R_{10} \\ I \end{bmatrix} = \begin{bmatrix} R_{10} \\ Si - O \end{bmatrix} = \begin{bmatrix} R_{10} \\ Si - R_{12} \end{bmatrix}$

R₉ is the direct bond; a straight-chain or branched C₁-C₄alkylene radical or a radical of the formula -c_{m,}H_{2m,}O-;

 R_{10} , R_{11} and R_{12} , independently of one another, are C_1 - C_{18} alkyl; C_1 - C_{18} alkoxy or a radical of the formula -0-0-0: R_{13} : $R_{$

R₁₃ is C₁-C₅alkyl;

m₁ is 1 to 4;

p₁ is 0 to 5;

A₁ is a radical of the formula

(2b)
$$\longrightarrow_{O-R_{14}}$$
; (2c) $-N \longrightarrow_{CO_2R_{15}}$; or of the formula

R₁₄ is hydrogen; C₁-C₁₀alkyl, -(CH₂CHR₁₆-O)_{n,}-R₁₅; or a radical of the formula

-CH₂-CH(-OH)-CH₂-O-T₁;

 R_{15} is hydrogen; M; C_1 - C_5 alkyl; or a radical of the formula $-(CH_2)_{m_2}$ - $O-(CH_2)_{m_3}$ -T, ;

R₁₆ is hydrogen; or methyl;

T₁ is hydrogen; or C₁-C₈alkyl;

Q₁ is C₁-C₁₈alkyl;

M is a metal cation;

m₂ and m₃, independently of one another, are 1 to 4; and

n₁ is 1 to 16.

Very particularly preferred triazine derivatives of the formula (2) correspond to the formulae

(2a)
$$OH_{18}$$
 OH_{18} OH_{18}

in which

 R_{17} and R_{18} , independently of one another, are C_3 - C_{18} alkyl; or -CH₂-CH(-OH)-CH₂-O-T₁; R_{19} is C_1 - C_{10} alkyl or a radical of the formula

(2a₁)
$$-CH_2$$
 or (2a₂) $-CH_2$ O- T_2 ;

R₂₀ is hydrogen; M; C₁-C₅alkyl; -NH-C₁-C₅alkyl; preferably -NH-tert-alkyl; or a radical of the formula -(CH₂)_m-O-T₂;

T₁ and T₂, indepenently of one another, are hydrogen; or C₁-C₅alkyl; and m is 1 to 4.

Of very particular interest are compounds of the formula (2a) and (2b) in which R_{17} and R_{18} , independently of one another, are C_1 - C_{18} alkyl; or - CH_2 -CH(-OH)- CH_2 -O- T_1 ; R_{19} is C_1 - C_{10} alkyl; and compounds of the formula (2c) and (2d) in which R_{17} and R_{18} , independently of one another, are C_1 - C_{18} alkyl or - CH_2 -CH(-OH)- CH_2 -O- T_1 ; and T_1 is hydrogen; or C_1 - C_5 alkyl.

Of the utmost interest are triazine compounds of the formula (2a) - (2d) in which R_{17} and R_{18} have the same meanings.

Further interesting triazine compounds which can be used according to the invention correspond to the formula

(3)
$$R_{23}$$
 R_{24} R_{22} R_{24} R_{24} R_{24} R_{25}

in which

R₂₁ is C₁-C₃₀alkyl; C₂-C₃₀alkenyl; unsubstituted C₅-C₁₂cycloalkyl or C₅-C₁₂cycloalkyl monoor polysubstituted by C₁-C₅alkyl; C₁-C₅alkoxy-C₁-C₁₂alkyl; amino-C₁-C₁₂alkyl; C₁-C₅monoalkylamino-C₁-C₁₂alkyl; C₁-C₅dialkylamino-C₁-C₁₂alkyl; a radical of the

formula (3a)
$$-(CH_2)\frac{1}{n_1}(O)\frac{1}{m_1}$$
; or (3b) ; in which

 R_{22} , R_{23} and R_{24} , independently of one another, are hydrogen, -OH; C_1 - C_{30} alkyl, C_2 - C_{30} alkenyl,

R₂₅ is hydrogen; or C₁-C₅alkyl;

m, is 0 or 1; and

n, is 1 to 5.

Preferred compounds correspond to the formula

$$R_{26}$$
 is $-O-CH_2-CH_{3-1}$; $-O-isoC_{18}H_{38}$; $-O-CH_2-CH_{3-1}$ $-O-n-C_{18}H_{37}$; or $-O-CH_2-CH_{3-1}$

-O-2-ethylhexyl; -O-(CH₂)₃-N(C₂H₅)₂; -O O
$$\longrightarrow$$
 ; -O \longrightarrow CH₃

$$-O-CH_{2}-C\overset{n-C_{12}H_{25}}{\underset{n-C_{10}H_{21}}{\leftarrow}};\quad -O-CH_{2}-C\overset{n-C_{8}H_{17}}{\underset{n-C_{6}H_{13}}{\leftarrow}};\quad -\overset{(CH_{2})_{r}-CH_{3}}{\underset{(CH_{2})_{s}-CH_{3}}{\leftarrow}}; \text{ and }$$

r and s, independently of one another, are 0 to 20.

Examples of triazine derivatives which can be used according to the invention correspond to the formulae

and also 2,4,6-tris(diisobutyl-4'-aminobenzalmalonate)-s-triazine and 2,4-bis(diisobutyl-4-aminobenzalmalonate)-6-(4'-aminobenzylidenecamphor)-s-triazine.

Likewise preferred triazine compounds which can be used according to the invention are described in EP-A-654469, for example the compound of the formula

According to the invention, particularly suitable triazine compounds are those described, for example, in EP-A-0,818450, for example the compound of the formula

Very particularly preferred triazine derivatives which can be used according to the invention correspond to the formula

R₂₇, R₂₈ and R₂₉, independently of one another, are a radical of the formula

(25c)
$$R_{31}$$
 R_{32} O OR_{30} ;

R₃₀ is hydrogen; alkali metal; an ammonium group -N(R₃₃)₄,

R₃₃ is hydrogen; C₁-C₅alkyl; or a polyoxyethylene radical which has 1 to 10 ethylene oxide units and the terminal OH group can be etherified with a C₁-C₅alcohol;

R₃₁ is hydrogen; -OH; or C₁-C₆alkoxy;

R₃₂ is hydrogen or -COOR₃₀; and

n is 0 or 1.

If R_{30} is alkali metal, this is in particular potassium or very particularly sodium. (R_{33})₄ is in particular a mono-, di- or tri- C_1 - C_4 alkylammonium salt, a mono-, di- or tri- C_2 - C_4 alkanol-ammonium salt or a C_1 - C_3 alkyl ester thereof.

If R_{33} is a C_1 - C_3 alkyl group, this is in particular a C_1 - C_2 alkyl group, in particular a methyl group, and if R_{33} is a polyoxyethylene radical, then the latter contains in particular 2 to 6 ethylene oxide units.

Preferred benzotriazole compounds which can be used according to the inv ntion correspond to the formula

T₁ is C₁-C₅alkyl or, preferably, hydrogen; and

 T_2 is C_1 - C_5 alkyl, preferably t-butyl, or phenyl-substituted C_1 - C_4 alkyl, in particular α, α -dimethylbenzyl.

A further preferred class of benzotriazole compounds which can be used according to the invention corresponds to the formula

T₂ is as defined in formula (26).

Other very particularly preferred benzotriazole compounds which can be used according to the invention correspond to the formula

(28)
$$N$$
 N N N N N , in which

T₂ is as defined in formula (26) and is preferably methyl, t-butyl or isooctyl.

Preferred vinyl-containing amides which can be used according to the invention correspond to the formula

(29)
$$R_{33}$$
-(Y)_m-CO-C(R_{34})=C(R_{35})-N(R_{36})(R_{37}), in which

R₃₃ is C₁-C₅alkyl, preferably methyl or ethyl, or unsubstituted phenyl or phenyl substituted by one, two or three of the radicals OH, C₁-C₅alkyl, C₁-C₅alkoxy or CO-OR₃₃;

R₃₄, R₃₅, R₃₆ and R₃₇, independently of one another, are C₁-C₅alkyl, preferably methyl or ethyl; or hydrogen;

Y is -NH or -O-; and

m is as defined above.

Preferred compounds of the formula (29) are 4-methyl-3-penten-2-one, ethyl 3-methyl-amino-2-butenoate, 3-methylamino-1-phenyl-2-buten-1-one and 3-methylamino-1-phenyl-2-buten-1-one.

Preferred cinnamides which can be used according to the invention correspond to the formula

(30)
$$R_{38}O - CH = CH - CO - NR_{39}R_{40}$$
, in which

R₃₈ is hydrogen or C₁-C₅alkoxy, preferably methoxy or ethoxy;

R₃₉ is hydrogen or C₁-C₅alkyl, preferably methyl or ethyl; and

R₄₀ is -(CONH)_m-phenyl, in which m is as defined above, and the phenyl group is unsubstituted or substituted by one, two or three of the radicals OH, C₁-C₃alkyl, C₁-C₃alkoxy or CO-OR₃₀.

R₄₀ is preferably phenyl, 4-methoxyphenyl or the phenylaminocarbonyl group.

Further preferred cinnamic acid derivatives are 2-ethylhexyl 4-methoxycinnamate or isoamylate or inter alia the cinnamic acid derivatives disclosed in US-A-5 601 811 and WO 97/00851.

Preferred sulfonated benzimidazoles which can be used according to the invention correspond to the formula

M is hydrogen or an alkali metal, preferably sodium, an alkaline earth metal, for example magnesium or calcium, or zinc.

Preferred Fischer base aldehydes which can be used according to the invention correspond to the formula

(32)
$$R_{41}$$
 R_{42} R_{42} R_{44} , in which

R₄₁ is hydrogen; C₁-C₅alkyl; C₁-C₁₈alkoxy; or halogen;

 R_{42} is C_1 - C_8 alkyl; C_5 - C_7 cycloalkyl; or C_6 - C_{10} aryl;

R₄₃ is C₁-C₁₈alkyl or a radical of the formula (32a)

R₄₄ is hydrogen; or a radical of the formula — c=0

 R_{45} is $\begin{bmatrix} R_{47} & R_{48} \\ N & C = 0 \end{bmatrix}$; C_1 - C_{18} alkoxy; or a radical of the formula

R₄₆ and R₄₇, independently of one another, are hydrogen; or C₁-C₅alkyl;

R₄₈ is hydrogen; C₁-C₅alkyl; C₅-C₇cycloalkyl; phenyl; phenyl-C₁-C₃alkyl;

 R_{49} is C_1 - C_{18} alkyl;

n is 0 or 1.

Further compounds which can be used with preference correspond to the formula

(33)
$$ZO_3S$$

$$R_{54}$$

$$C_m - C_n R_{53}$$

$$R_{54}$$

$$R_{53}$$

$$R_{54}$$

$$R_{55}$$

$$R_{51}$$

$$R_{51}$$

$$X_1$$

R₅₀, R₅₁, R₅₂, R₅₃, R₅₄, independently of one another, are hydrogen, C₁-C₈alkyl or C₅-C₁₀cycloalkyl;

R₅₅ is hydrogen; C₁-C₈alkyl; C₅-C₁₀cycloalkyl; hydroxyl; C₁-C₈alkoxy; COOR₅₆; or CONR₅₇R₅₈;

 $R_{56},\,R_{57}$ and $R_{58},$ independently of one another, are hydrogen or $C_1\text{-}C_6\text{alkyl};$

X and Y, independently of one another, are hydrogen, -CN; CO_2R_{59} ; $CONR_{59}R_{60}$; or COR_{59} ; where the radicals X and Y may additionally be a C_1 - C_8 alkyl radical, a C_5 - C_{10} alkyl radical, in particular phenyl, or a heteroaryl radical having 5 to 6 ring atoms, where, in addition, X and Y or

R₅₀ together with one of the radicals X and Y can represent the radical to complete a 5- to 7-membered ring which may contain up to 3 heteroatoms, in particular oxygen and/or nitrogen, where the ring atoms may be substituted, in particular by exocyclically double-bonded oxygen (keto oxygen) and/or C₁-C₈alkyl and/or C₅-C₁₀cycloalkyl radicals, and/or may contain C=C double bonds;

Z is hydrogen; ammonium; alkali metal ion; in particular lithium, sodium, potassium, 1/2 equivalents of alkaline earth metal ion, preferably calcium, magnesium or the cation of an organic nitrogen base used to neutralize the free acid group,

R₅₉ and R₆₀, independently of one another, are hydrogen, C₁-C₈alkyl or C₅-C₁₀cycloalkyl; and

n and m, independently of one another, are 0 or 1.

Preferred diphenylmalonitriles which can be used according to the invention correspond to the formula

 R_{61} and R_{62} , independently of one another, are C_1 - C_{12} alkyl; or C_1 - C_{12} alkoxy; and n is 0-3.

Other organic UV filters which can be used according to the invention correspond to the formula

R₆₃ and R₆₄, independently of one another, are C₁-C₅alkyl, in particular ethyl.

Other preferred chemical compound classes of UV filters which can be used according to the invention are:

 p-aminobenzoic acid derivatives (PABA), in particular 2-ethylhexyl 4-dimethylaminobenzoate;

- salicylic acid derivatives, in particular 2-ethylhexyl salicylates; homosalates; and isopropyl salicylates;
- benzophenone derivatives, in particular benzophenone-2, -3, and -4;
- dibenzoylmethane derivatives, in particular 1-(4-tert-butylphenyl)-3-(4-methoxyphenyl)-propane-1,3-dione or butylmethoxydibenzoylmethane;
- diphenylacrylates, in particular 2-ethylhexyl 2-cyano-3,3-diphenylacrylate, ethyl 2-cyano-3,3'-diphenylacrylate and 3-(benzofuranyl) 2-cyanoacrylate;
- 3-imidazol-4-ylacrylic acid and 3-imidazol-4-yl acrylate;
- benzofuran derivatives, in particular the p-aminophenylbenzofuran derivatives published in EP-A-582,189, US-A-5,338,539 and US-A-5-518,713;
- camphor derivatives, in particular 3-(4'-methyl)benzylidenebornan-2-one, 3-benzylidenebornan-2-one, N-[2(and 4)-2-oxyborn-3-ylidenemethyl)benzyl]acrylamide polymer, 3-(4'-trimethylammonium)benzylidenebornan-2-one methylsulfate, 3,3'-(1,4-phenylenedimethine)-bis(7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-methanesulfonic acid) and salts thereof, 3-(4'-sulfo)benzylidenebornan-2-one and salts thereof; and
- menthyl o-aminobenzoate.

The UV filters listed above can be used according to the invention as individual compounds or also, preferably, as mixtures.

Preference is given to using the following mixtures of organic UV filters:

- mixtures of methylenebisbenzotriazolyltetramethylbutylphenol and octyltriazone;
- mixtures of octyltriazone and methylenebisbenzotriazolyltetramethylbutylphenol;
- mixtures of 2-[(2,4-methoxy)phenyl]-4,6-bis[(2-hydroxy-4-methoxy)phenyl]- (1,3,5)triazine and methylenebisbenzotriazolyltetramethylbutylphenol;
- mixtures of methylenebisbenzotriazolyltetramethylbutylphenol and dioctylbutamidotriazone;
- mixtures of methylenebisbenzotriazolyltetramethylbutylphenol and octyl-2,2'- methylenebis[6-(2H-benzotriazol-2-yl)-4-methylphenol,
- mixtures of octyltriazone and trisresorcinyltriazine;

mixtures of 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-methylphenol, octyltriazone

mixtures of 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-methylphenol, octyltriazone

mixtures of methylenebisbenzotriazolyltetramethylbutylphenol, octyltriazone and the

- mixtures of methylenebisbenzotriazolyltetramethylbutylphenol and the compound

mixtures of methylenebisbenzotriazolyltetramethylbutylphenol,
 dioctylbutamidotriazone and the compound of the formula (37).

In the radicals defined above, C₁-C₁₈alkyl are straight-chain or branched alkyl radicals, for example methyl, ethyl, n-propyl, isopropyl, n-butyl, sec-butyl, tert-butyl, amyl, isoamyl or tert-amyl, heptyl, octyl, isooctyl, nonyl, decyl, undecyl, dodecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl or octadecyl.

C₁-C₁₈Alkoxy radicals are straight-chain or branched alkyl radicals, for example methoxy, ethoxy, n-propoxy, isopropoxy, n-butoxy, sec-butoxy, tert-butoxy, amyloxy, isoamyloxy or tert-amyloxy, heptyloxy, octyloxy, isooctyloxy, nonyloxy, decyloxy, undecyloxy, dodecyloxy, tetradecyloxy, pentadecyloxy, hexadecyloxy, heptadecyloxy or octadecyloxy.

C₂-C₁₈Alkenyl is, for example, allyl, methallyl, isopropenyl, 2-butenyl, 3-butenyl, isobutenyl, n-penta-2,4-dienyl, 3-methylbut-2-enyl, n-oct-2-enyl, n-dodec-2-enyl, isododecenyl, n-dodec-2-enyl or n-octadec-4-enyl.

The mixtures of micronized organic UV filters which can be used according to the invention can be prepared in different ways.

Firstly, at least two of the abovementioned organic UV filters can be mixed as individual substances in the preparation process of the microparticles (micronization).

Another preparation option involves thoroughly mixing the already micronized individual substances of the UV filters together.

A third preparation option involves melting together at least two of the abovementioned UV filters. Cooling the melt produces a homogeneous composite, which is micronized in the usual manner.

The homogeneous composites of at least two organic UV filters are also provided by the invention.

The invention further provides composites obtainable by fusing one or more inorganic micropigments into one or more organic UV filters.

Examples of micropigments are, for example, TiO₂, ZnO, iron oxides or other inorganic oxides, mica or other suitable inorganic minerals, and also Ti, alkaline earth metal or zinc salts of organic acids.

In so doing, the undesired photocatalytic properties of some of these inorganic micropigments (TiO₂, ZnO) can be simultaneously suppressed, and their positive properties can also be fully utilized.

The abovementioned inorganic UV filters are advantageously fused into methylenebisbenzotriazolyltetramethylbutylphenol. The resulting composite is then micronized in the usual manner.

The invention further provides composites obtainable by melting at least two electrically neutral organic UV filters with cationically or anionically charged compounds.

For this, cationically or anionically charged compounds are melted with the corresponding organic, electrically neutral UV filters and then cooled. This process permits, in the subsequent micronization step, the preparation of organic UV filter pigments having a permanent finishing of a positive or negative charge. Such a finishing effectively prevents aggregation of the micronized particles in the sunscreen preparations which can occur in cases where the particle diameter is < $1\mu m$. An otherwise customary "coating" of these particles having a repelling effect then sometimes becomes superfluous.

Cationically or anionically charged compounds which can be used are UV filters and also other compounds which have one or more cationic or anionic groups, for example

- N,N,N-trimethyl-4-(2-oxoborn-3-ylidenemethyl)aniline methylsulfate;
- camphorbenzalkonium methosulfate;
- fatty amines;
- betaines, for example cocamidopropylbetaine;
- quats, for example ricinoleamidopropyltrimonium methosulfate, Quarternium 18, or cetyltrimethylammonium bromide;
- behenic acid and other organic acids, for example isostearic acid, citricmonoglyceride or sodium methyl cocoyl taurate;
- phospholipids, for example phosphatidylcholine, phosphatidylserine or alkylamine oxide;
- ceramides and pseudoceramides and phytosterols.

The last-named compounds impart an oleophobic finishing to the micronized UV filters.

The proportion of cationic or anionic compounds in the composite is between 0.001 and 5% by weight, preferably 0.01 to 3% by weight, based on the weight of the UV filter(s).

The invention further provides composites obtainable by melting at least one sparingly soluble or insoluble organic UV filter with antioxidants.

For this, the sparingly soluble or insoluble organic UV filter(s) is/are melted together with antioxidants, cooled and then micronized in the usual manner.

Suitable antioxidants which can be used according to the invention are all organic substances having scavenger properties which can be melted together with organic UV filters. This gives novel types of micropigments which simultaneously prevent tanning of the skin and offer antioxidative action on its surface. This property is desired for cosmetic sun protection since, under the influence of UV and light, harmful free radicals can be formed both in formulations and on the skin. These can, for example, lead to so-called Mallorca acne or to premature skin ageing. By finishing the micronized UV filters with antioxidants, not only is protection against UV damage and prevention of tanning achieved, but also protection against photochemical degradation of constituents in the sunscreen formulation.

The proportion of antioxidants in the composite is generally between 0.001 and 30% by weight, preferably 0.01 to 3% by weight, based on the weight of the UV filter(s).

A content of antioxidants is particularly advantageous in micropigments which, in addition to organic UV filters, comprise the abovementioned photocatalytically active inorganic micropigments, for example titanium dioxide, zinc oxide (including coated) or other suitable inorganic oxides, for example iron oxide.

Examples of antioxidants which may be listed are the following compounds:

- tocopherols, for example α-tocopherol (CAS 59-02-9), tocopheryl acetate, vitamin E succinate,

- N-butylated hydroxytoluene (BHT; CAS 128-37-0);
- butylated hydroxyanisole (BHA);

- 2,4,6-tris(3,5-di-t-butyl-4-hydroxybenzyl)mesitylene (CAS 1709-70-2)

- tetrakis[methylene-3(3',5'-di-t-butyl-4'-hydroxyphenyl)propionate]methane (CAS 6683-19-8);

- compound of the formula

- vanillin;
- ubiquinone;
- ferulic acid and derivatives;
- rutic acid and derivatives;
- urocanic acid and derivatives; and
- propolis.

Preference is given to using the following mixtures of antioxidants and organic UV filters:

- mixtures of methylenebisbenzotriazolyltetramethylbutylphenol, octyltriazone, titanium dioxide and tocopherol,
- mixtures of 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-methylphenol, octyltriazone, trisresorcinyltriazine and vitamin E

mixtures of methylenebisbenzotriazolyltetramethylbutylphenol, octyltriazone

The invention further provides composites obtainable by fusing meltable cosmetic, vegetable and pharmaceutical active ingredients into organic UV filters.

In general, micronized UV filters can be used as carriers of highly active substances, in particular cosmetic and/or pharmaceutical active substances. The advantage of such composites lies in the fact that it is possible for them to release the active substance(s) from the solid (slow release). A slow release also guarantees the uniform effectiveness of highly active active ingredients, for example antiinflammatories, care active ingredients or trace elements, for example Zn²⁺ or Mg²⁺, over the entire useful life of the UV pigments.

Examples of active ingredients which can be used and which may be mentioned are:

- active ingredients for antimicrobial finishing and simultaneous antiinflammatory action,
 for example triclosan or diclosan;
- antiinflammatory active ingredients, for example farnesol, panthenol or avocado oil;
- active ingredients having a deodorant or antiperspirant action, for example Zn ricinoleates and alkyl citrates,
- undecylenic acid and derivatives thereof (e.g. diethanolamides)
- zinc undecylate;
- pyrithiones, for example sodium pyrithione;

- fused-in fragrances or fragrance mixtures, for example menthol, geraniol etc., which impart a permanent odour which is uniform in intensity to these micropigments and the formulations which comprise them.

To prepare the micronized organic UV filters or the micropigment mixtures, it is possible to use all known processes which are suitable for the preparation of microparticles, for example:

- wet grinding with a hard grinding medium, for example zirconium silicate and a protective surfactant or a protective polymer in water or a suitable organic solvent;
- spray drying from a suitable solvent, for example aqueous or organic suspensions containing solvent, or true solutions in water, ethanol, dichloroethane, toluene, Nmethylpyrrolidone etc.;
- by expansion of supercritical liquids (e.g. CO₂) in accordance with the RESS process (Rapid Expansion of Supercritical Solutions) in which the UV filter(s) is/are dissolved or expansion of liquid carbon dioxide together with a solution of one or more UV filters in a suitable organic solvent;
- by reprecipitation from suitable solvents, including supercritical liquids (GASR process = Gas Anti-Solvent Recrystallization / PCA process = Precipitation with Compressed Antisolvents).

Grinding apparatuses which can be used for the preparation of the micronized organic UV absorbers according to the invention are, for example, a jet, ball, vibratory or hammer mill, preferably a high-speed stirred mill. Grinding preferably takes place using a grinding auxiliary, for example an alkylated vinylpyrrolidone polymer, a vinylpyrrolidone/vinyl acetate copolymer, an acyl glutamate, an alkyl polyglucoside, ceteareth-25 or, in particular, a phospholipid.

The resulting micropigments or mixtures of micropigments usually have an average particle size of from 0.02 to 2 nm, preferably 0.05 to 1.5 nm, and very particularly from 0.1 to 1.0 nm.

Because of the ir lipophilicity, they can, alone or together with other soluble organic UV absorbers, be readily incorporated into oil- and fat-containing cosmetic formulations, for example oils, O/W or W/O emulsions, wax pencils or gels, by known methods.

Surprisingly, formulations are obtained which have equal or improved protective action using less or even no soluble UV absorbers.

The invention further provides a cosmetic formulation comprising a mixture of micropigments, if desired one or more antioxidants and/or inorganic pigments and/or a cationic or anionic compound, and cosmetically compatible carriers or auxiliaries.

Cosmetic formulations according to the invention include various cosmetic compositions. In particular, the following compositions are, for example, suitable:

- skincare compositions, for example skin washes and cleansers in the form of bar or liquid soaps, syndets or washing pastes,
- bath preparations, for example liquid (foam baths, milks, shower preparations) or solid bath preparations, for example bath tablets or bath salts;
- skincare compositions, for example skin emulsions, multiple emulsions or skin oils;
- decorative bodycare compositions, for example face make-up in the form of day
 creams or powder creams, face powder (loose or pressed), blusher or cream make-up,
 eyecare compositions, for example eyeshadow preparations, mascara, eyeliner, eye
 creams or eye-fix creams; lipcare compositions, for example lipstick, lip gloss, lip liner
 pencil, nailcare compositions, such as nail varnish, nail varnish remover, nail hardeners
 or cuticle removers;
- personal hygiene care compositions, for example personal hygiene washing lotions or personal hygiene sprays;
- footcare compositions, for example foot baths, foot powders, foot creams or foot balsams, special deodorants and antiperspirants or products for removing calluses;
- light protection compositions, such as sun milks, lotions, creams and oils, sun blocks or tropicals, pretanning preparations or aftersun preparations;
- skin tanning compositions, for example self-tanning creams;
- depigmentation products, for example preparations for skin bleaching or compositions for skin lightening;

- insect-repelling compositions ("repellents"), for example insect oils, lotions, sprays or sticks;
- deodorants, such as deodorant sprays, pump sprays and deodorant gels, sticks or roller balls;
- antiperspirants, for example antiperspirant sticks, creams or roller balls;
- compositions for cleansing and caring for blemished skin, for example syndets (solid or liquid), peeling or exfoliation preparations or peeling masks;
- depilatories in chemical form, for example depilatory powders, liquid depilatories, cream or paste depilatories, depilatories in gel form or aerosol foams;
- shaving compositions, for example shaving soap, foaming shaving creams, nonfoaming shaving creams, foams, gels, preshave preparations for dry shaving, aftershaves or aftershave lotions;
- fragrances, for example fragrance water (eau de Cologne, eau de toilette, eau de parfum, parfum de toilette, parfum), perfume oils or perfume creams;
- compositions for dental, denture and mouth care, for example toothpastes, gel toothpastes, tooth powders, mouthwash concentrates, antiplaque mouthrinses, denture cleaners or denture adhesives;
- cosmetic compositions for treating hair, for example hair cleansers in the form of shampoos, hair conditioners, haircare compositions, for example pretreatment compositions, hair tonic, styling creams, styling gels, pomades, hair rinses, treatment packs, intensive hair treatments, compositions for shaping hair, for example waving agents for the preparation of permanent waves (hotwave, mildwave, coldwave), hair-smoothing preparations, liquid hair-setting compositions, hair mousses, hair sprays, bleaching agents, for example hydrogen peroxide solutions, lightening shampoos, bleaching creams, bleaching powders, bleaching pastes or oils, temporary, semipermanent or permanent hair colorants, preparations containing self-oxidizing dyes, or natural hair colorants, such as henna or camomile.

These listed end formulations can be in the form of various application forms, for example

- in the form of liquid preparations as a W/O, O/W, O/W/O, W/O/W, PIT and all other types of microemulsions,
- in the form of a gel,
- in the form of an oil, a cream, milk or lotion.
- in the form of a powder, a lacquer, a tablet or make-up,

- in the form of a stick,
- in the form of a spray (spray with propellant or pump spray) or an aerosol,
- in the form of a foam, or
- in the form of a paste.

The cosmetic formulations according to the invention can advantageously comprise further substances which absorb UV radiation in the UVB region. The total amount of filter substances here is 0.1 to 30% by weight, preferably 0.5 to 10% by weight, in particular 1 to 6% by weight, based on the total weight of the composition.

In particular, suitable additional UVB filters are oil-soluble, nonmicronized compounds, for example organic UV absorbers from the class of p-aminobenzoic acid derivatives, salicylic acid derivatives, benzophenone derivatives, dibenzoylmethane derivatives, diphenylacrylate derivatives, benzofuran derivatives, polymeric UV absorbers, comprising one or more organosilicon radicals, cinnamic acid derivatives, camphor derivatives, trianilino-s-triazine derivatives, phenylbenzimidazolesulfonic acid and salts thereof, menthyl anthranilate, benzotriazole derivatives, and/or an inorganic micropigment chosen from zinc oxide, mica or TiO₂ coated with aluminium oxide or silicon dioxide.

Examples of compounds of p-aminobenzoic acid derivatives:
 4-aminobenzoic acid (PABA); ethyldihydroxypropyl-PABA of the formula

$$H(O-CH_2CH_2)n$$
 $N-CO-O-(CH_2CH_2-O)x-C_2H_5$, in which m, n and x have the same

meaning and are each at most 25;

Examples of compounds of salicylic acid derivatives:

homomenthyl salicylate of the formula

salicylate of the formula

benzoate of the formula (10) (CH₃)₂N—COO-amyl ; octyl salicylate of the

formula O-isooctyl ; or 4-isopropylbenzyl salicylate of the

- Examples of compounds of benzophenone derivatives:
 benzophenone-3-(2-hydroxy-4-methoxybenzophenone), benzophenone-4-(2-hydroxy-4-methoxybenzophenone-5-sulfonic acid) or benzophenone-8-(2,2'-dihydroxy-4-methoxybenzophenone).
- Examples of compounds of dibenzoylmethane derivatives:
 butylmethoxydibenzoylmethane[1-(4-tert-butyl)-3-(4-methoxyphenyl)propane-1,3-dione].
- Examples of compounds of diphenylacrylate derivatives: octocrylene 2-ethylhexyl-2-cyano-3,3'-diphenylacrylate or etocrylene ethyl-2-cyano-3,3'-diphenylacrylate.
- Examples of compounds of benzofuran derivatives:
 3-benzofuranyl 2-cyanoacrylate, 2-(2-benzofuranyl)-5-tert-butylbenzoxazole or 2-(p-aminophenyl)benzofuran and, in particular, the compound of the formula

- Examples of compounds of polymeric UV absorbers which comprise one or more organosilicon radicals:

benzylidenemalonate derivatives, in particular the compound of the formula

in which

R₂₄ is hydrogen or methoxy and

r is approximately 7; the compound of the formula

$$\begin{array}{c} \text{O-Si(CH}_3)_3\\ \text{O-Si-CH}_3\\ \text{O-Si(CH}_3)_3\\ \text{CH}_3\\ \end{array}; \text{ or }$$

$$\begin{array}{c} \text{O-Si(CH}_3)_3 \\ \text{OH} \\ \text{OH} \\ \text{CH}_3 \\ \text{CH}_3 \end{array}$$

Examples of compounds of cinnamic esters:

octyl methoxycinnamate (2-ethylhexyl 4-methoxycinnamate), diethanolamine
methoxycinnamate (diethanolamine salt of 4-methoxycinnamic acid), isoamyl p-

methoxycinnamate (2-isoamyl 4-ethoxycinnamate), 2,5-diisopropyl methylcinnamate or a cinnamic acid amido derivative.

- Examples of compounds of camphor derivatives: 4-methylbenzylidenecamphor [3-(4'-methyl)benzylidenebornan-2-one], 3-benzylidenecamphor (3-benzylidenebornan-2-one), polyacrylamidomethylbenzylidenecamphor {N-[2(and 4)-2-oxyborn-3-ylidene-methyl)benzyl]acrylamide polymer}, trimoniumbenzylidenecamphor sulfate [3-(4'-trimethylammonium)benzylidenebornan-2-one methylsulfate], terephthalylidenedicamphorsulfonic acid {3,3'-(1,4-phenylenedimethine)bis(7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-methanesulfonic acid} or salts thereof, or benzylidenecamphorsulfonic acid [3-(4'-sulpho)benzylidenebornan-2-one] or salts thereof.
- Examples of compounds of trianilino-s-triazine derivatives: octyltriazine[2,4,6-trianilino(p-carbo-2'-ethyl-1'-oxy)-1,3,5-triazine, and the trianilino-s-triazine derivatives described in US-A-5,332,568, US-A-5,252,323, WO 93/17002 and WO 97/03642 and EP-A-0,517,104.
- Examples of compounds of benzotriazoles:
 2-(2-hydroxy-5-methylphenyl)benzotriazole.

The examples below serve to illustrate the invention without limiting it thereto. The cosmetic active substances are primarily given with their INCI name (INCI = International Nomenclature of Cosmetic Ingredients).

Example 1:

50 parts of Methylene Bis-benzotriazolyl Tetramethylbutylphenol and 50 parts of Octyl Triazone are ground together using a grinding medium of zirconium silicate sand, a protective surfactant (Alkyl Polyglucoside) and water in a bead mill to give a mixed micropigment having a d_{50} of 190 nm. After the grinding medium has been separated off, the suspension of the mixed micropigment can be used to prepare sunscreen formulations.

Example 2:

32 parts of Octyl Triazone, 1 part of cetyltrimethylammonium bromide and 66 parts of Methylene Bis-benzotriazolyl Tetramethylbutylphenol are homogeneously melted together. The mixture is rapidly cooled to room temperature, and the solidified melt is comminuted mechanically (beater mill). This resulting powder is slurried in water, Decyl Glycoside is added, and the mixture is micronized together with a grinding auxiliary ('heavy sand') to a particle size diameter d_{50} of 200 nm. After the grinding auxiliary has been removed, an aqueous suspension of the micronized UV absorber composite is obtained. This suspension is rendered slightly acidic with citric acid and can be used for the preparation of cosmetic and pharmaceutical formulations.

Example 3:

25 parts of 2-[(2,4-methoxy)phenyl]-4,6-bis[(2-hydroxy-4-methoxy)phenyl]-(1,3,5)-triazine, 74 parts of Methylene Bis-benzotriazolyl Tetramethylbutylphenol and 1 part of Tetrakis[methylene-3(3',5'-di-t-butyl-4'-hydroxyphenyl)propionate]methane are homogeneously fused together. The mixture is rapidly cooled to room temperature, and the solidified melt is comminuted mechanically (beater mill). This resulting powder is slurried in water, firstly Decyl Glycoside is added, then, after continued grinding, Ceteareth-25, and the mixture is micronized together with a grinding auxiliary ('heavy sand') to a particle size diameter d₅₀ of 190 nm. After the grinding auxiliary has been separated off, an aqueous suspension of the micronized UV absorber composite is obtained, which can be used for the preparation of cosmetic and pharmaceutical formulations.

Example 4:

25 parts of Dioctyl Butamido Triazone are dissolved in 75 parts of molten Methylene Bisbenzotriazolyl Tetramethylbutylphenol. The mixture is cooled rapidly, comminuted mechanically to give a fine powder and then ground with a grinding medium of zirconium silicate sand, a protective surfactant (phospholipid) and water to give a micropigment having a d₅₀ of 300 nm. The micropigment suspension separated off from the grinding medium is used for the preparation of sunscreen formulations.

Example 5:

24 parts of Octyl Triazone, 5 parts of Titanium Dioxide and one part of Tocopherol are mixed into 70 parts of molten Methylene Bis-benzotriazolyl Tetramethylbutylphenol. The mixture is cooled rapidly, comminuted mechanically to give a fine powder and then ground with a grinding medium of zirconium silicate sand, a protective surfactant (Alkyl Polyglucoside) and water to give a micropigment. The micropigment suspension separated off from the grinding medium is used for the preparation of sunscreen formulations.

In Examples 6 to 11 below, suspensions of microcomposites having the following compositions are prepared analogously to Examples 1 and 2:

Example 6:

60 parts of 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-methylphenol, 20 parts of Octyl Triazone, 19 parts of Tris Resorcinyl Triazine and 1 part of vitamin E, adjusted to pH 6.5 with citric acid.

Example 7:

60 parts of 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-methylphenol, 20 parts of Octyl Triazone and 20 parts of the compound of the formula

Example 8:

59 parts of 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-methylphenol, 20 parts of Octyl Triazone,

20 parts of the compound of the formula (102)

and adjusted to pH 6.5 with citric acid.

Example 9:

75 parts of Methylene Bis-benzotriazolyl Tetramethylbutylphenol, 10 parts of Octyl Triazone (grinding at pH < 5, adjusted with citric acid),

14 parts of the compound of the formula (103)

and

1 part of the compound of the formula

Example 10:

80 parts of Methylene Bis-benzotriazolyl Tetramethylbutylphenol, and

20 parts of the compound of the formula (104)

Example 11:

50 parts of Methylene Bis-benzotriazolyl Tetramethylbutylphenol,

10 parts of Dioctyl Butamido Triazone (grinding at pH < 5, adjusted to pH 6.5 with citric acid) and

20 parts of the compound of the formula (102).

Example 12: O/W lotion for preventing tanning

		<u>%</u>
Α	Polyglyceryl-3 Methylglucose Distearate	2.0
•	Decyl Oleate	5.7
	Isopropyl Palmitate	6.0
	Caprylic/Capric Triglyceride	7.5
В	Glycerin	3.0
	Phenonip	0.5
	Water	69.3
C	Carbomer	0.2
	Isopropyl Palmitate	0.8
D	Micropigment from Example 2	5.0
Ε	NaOH (10%)	as required
<u>Exar</u>	nple 13: O/W Emulsion	
		<u>%</u>
Pota	assium Cetyl Phosphate	2.00
	ontanyl PVP	1.00
	rylic/Capric Triglyceride	5.00
	earyl Isononanoate	5.00
	2-15 Alkyl Benzoate	5.00
	ceryl Stearate	3.00
	yl Alcohol	1.00
	noxyethanol&Parabens	1.00
Octyl Methoxycinnamate		5.00
	ethicone	0.10
	onized Water	64.15 0.10
	bomer (Carbopol 981)	3.00
	cerin DH (10%)	1.00
	ropigment from Example 1	4.00
IVIIC	opiginont nom Example i	7.00

Example 14: O/W Emulsion:

Cetearyl Alcohol & Dicetyl Phosphate & Ceteth-10 Phosphate Caprylic/Capric Triglyceride Cetearyl Isononanoate C12-15 Alkyl Benzoate Phenoxyethanol & Parabens Octyl Methoxycinnamate Dimethicone	% 6.00 5.00 5.00 5.00 1.00 5.00 0.20
Deionized Water Carbomer (Carbopol 981) Glycerin NaOH (10%)	64.70 0.10 3.00 0.65
Micropigment from Example 3	4.00
Example 15: O/W Emulsion: Isopropyl myristate & Trilaureth-4 Phosphate Tricontanyl PVP Caprylic/Capric Triglyceride Cetearyl Isononanoate C12-15 Alkyl Benzoate Glyceryl Stearate Cetyl Alcohol Phenoxyethanol & Parabens Octyl Methoxycinnamate Dimethicone	% 5.00 1.00 5.00 2.00 5.00 2.00 1.00 1.00 5.00 0.10
Deionized Water Carbomer (Carbopol 981) Glycerin NaOH (10%)	66.30 0.10 3.00 0.50
Micropigment from Example 4	4.00

Example 16: O/W Emulsion

	<u>%</u>
Sodium Stearyl Lactate Tricontanyl PVP	1.50
Tricontanyl PVP	1.00
Caprylic/Capric Triglyceride	5.00
Cetearyl Isononanoate	5.00
C12-15 Alkyl Benzoate	5.00
Glyceryl Stearate	3.50
Cetyl Alcohol	2.00
Phenoxyethanol & Parabens	1.00
Octyl Methoxycinnamate	5.00
Dimethicone	0.20
Deionized Water	63.60
Carbomer (Carbopol 981)	0.10
Glycerin	3.00
NaOH (10%)	0.10
Micropigment from Example 6	4.00
Example 17: O/W Emulsion	
	<u>%</u>
Cetearyl Alcohol & Sodium Cetearyl Sulfate	5.00
Caprylic/Capric Triglyceride	5.00
Cetearyl Isononanoate	5.00
C12-15 Alkyl Benzoate	5.00
Phenoxyethanol & Parabens	1.00
Octyl Methoxycinnamate	5.00
Dimethicone	0.10
Deionized Water	65.90
Glycerin	3.00
NaOH (10%)	0.30
Micropigment from Example 9	4.00
more pigment from Example 5	4.00

Example 18: O/W Emulsion

	<u>%</u>
Lauryl Glucoside & Polyglyceryl-2 Dihydroxystearate & Glycerin Tricontanyl PVP Caprylic/Capric Triglyceride Cetearyl Isononanoate C12-15 Alkyl Benzoate Glyceryl Stearate Cetyl Alcohol Phenoxyethanol & Parabens Octyl Methoxycinnamate Dimethicone	3.00 1.00 4.00 4.00 5.00 2.00 3.00 1.00 5.00 0.20
Deionized Water Carbomer (Carbopol 981) Glycerin NaOH (10%)	64.49 0.10 3.00 0.21
Micropigment from Example 8	4.00
Example 19: O/W Emulsion:	
Cetaryl Glucoside & Cetearyl Alcohol Tricontanyl PVP Caprylic/Capric Triglyceride Cetearyl Isononanoate C12-15 Alkyl Benzoate Phenoxyethanol&Parabens Octyl Triazone 4-Methylbenzylidene camphor Dimethicone	% 4.50 1.00 5.00 5.00 5.00 1.00 3.00 3.00 0.20
Deionized Water Steareth-10 Allyl Ether/Acrylates Copolymer Glycerin NaOH (10%)	64.65 5.00 3.00 1.00
Micropigment from Example 2	4.00

Example 20: O/W Emulsion

	<u>%</u>
Cetearyl Glucoside	5.00
Tricontanyl PVP	1.00
Caprylic/Capric Triglyceride	5.00
Cetearyl Isononanoate	5.00
C12-15 Alkyl Benzoate	5.00
Phenoxyethanol & Parabens	1.00
Octocrylene	3.00
Octyl Methoxycinnamate	4.00
Dimethicone	0.20
Deionized Water	63.15
Carbomer (Carbopol 981)	0.50
Glycerin	3.00
NaOH (10%)	0.15
Micropigment from Example 2	4.00
Example 21: O/W Emulsion:	
	<u>%</u>
Polyglyceryl-10 Petastearate & Behenyl Alcohol & Sodium	2.50
Stearoyl Laurate	
Caprylic/Capric Triglyceride	5.00
Cetearyl Isononanoate	5.00
C12-15 Alkyl Benzoate	5.00
Glyceryl Stearate	3.00
Cetearyl Alcohol	2.00
Phenoxyethanol&Parabens	1.00
Octyl Methoxycinnamate	5.00
Dimethicone	0.20
Deionized Water	64.75
Carbomer (Carbopol 981)	0.15
Glycerin	3.00
NaOH (10%)	0.40
Micropigment from Example 9	4.00

Example 22: O/W Emulsion:

	<u>%</u>
Palmitic Acid & Stearic Acid	1.80
Glyceryl Stearate SE	3.00
Tricontanyl PVP	1.00
Caprylic/Capric Triglyceride	5.00
Cetearyl Isononanoate	5.00
C12-15 Alkyl Benzoate	5.00
Glyceryl Stearate	0.50
Phenoxyethanol & Parabens	1.00
Octyl dimethyl PABA	5.00
Dimethicone	0.10
Deionized Water	64.15
Carbomer (Carbopol 981)	0.10
Glycerin	3.00
NaOH (10%)	0.50
Micropigment from Example 1	4.00
Example 23: O/W Emulsion:	
	<u>%</u>
Glyceryl Stearate & PEG 100 Stearate	3.00
Tricontanyl PVP	1.00
Caprylic/Capric Triglyceride	5.00
Cetearyl Isononanoate	5.00
C12-15 Alkyl Benzoate	5.00
Cetearyl Alcohol	3.00
Phenoxyethanol&Parabens	1.00
Octyl Methoxycinnamate	5.00
Dimethicone	0.10
Deionized Water	64.60
Carbomer (Carbopol 981)	0.10
Glycerin	3.00
NaOH (10%)	0.20
Micropigment from Example 3	4.00

Example 24: O/W Emulsion:

	<u>%</u>
Steareth-2	2.50
Steareth-21	1.00
Tricontanyl PVP	1.00
Caprylic/Capric Triglyceride	5.00
Cetearyl Isononanoate	5.00
C12-15 Alkyl Benzoate	5.00
Cetyl Alcohol	1.00
Phenoxyethanol & Parabens	1.00
Methyl Anthranilate	3.00
Octyl Methoxycinnamate	4.00
Dimethicone	0.10
Deionized Water	63.95
Carbomer (Carbopol 981)	0.20
Glycerin	3.00
NaOH (10%)	0.25
Micropigment from Example 4	4.00
Example 25: O/W Emulsion:	
	<u>%</u>
Channel Stranger Cotonath 20 9 Cotonath 10 9 Cotonal Alachai	
Glyceryl Stearate&Cetareth-20 & Cetareth-12 & Cetaryl Alcohol	5.00
& Cetyl Palmitate Tricontanyl PVP	1.00
Caprylic/Capric Triglyceride	5.00
Cetearyi Isononanoate	5.00
C12-15 Alkyl Benzoate	5.00
Phenoxyethanol & Parabens	1.00
4-Methylbenzylidene camphor	5.00
Dimethicone	0.10
Deionized Water	65.60
Carbomer (Carbopol 981)	0.10
Glycerin	3.00
NaOH (10%)	0.20
Micropigment from Example 3	4.00

Example 26: O/W Emulsion

Octyldecyl Phosphate Tricontanyl PVP Caprylic/Capric Triglyceride Cetearyl Isononanoate C12-15 Alkyl Benzoate Phenoxyethanol & Parabens Octyl methoxycinnamate Dimethicone	% 3.00 1.00 5.00 5.00 5.00 1.00 5.00 0.10
Deionized Water Sodium Cocoyl Glutamate Steareth-10 Allyl Ether/Acrylates Copolymer Glycerin NaOH (10%) Micropigment from Example 4 Example 27: O/W Emulsion:	64.50 0.60 0.50 3.00 2.30 4.00
	<u>%</u>
Polyglyceryl-3 Methyl Glucose Distearate Tricontanyl PVP Tocopherol&Ascorbyl Palmitate & Ascorbic Acid&Citric Acid &	2.00 1.00 0.05
PEG-8 Decyl Oleate Isopropyl Palmitate Caprylic/Capric Triglyceride Glyceryl Stearate Cetearyl Alcohol 2-[(2,4-Methoxy)phenyl]-4,6-bis[(2-hydroxy-4-methoxy)phenyl]-(1,3,5)triazine	4.50 6.00 5.00 1.00 1.00 2.00
Octyl Methoxycinnamate Deionized Water Phenoxyethanol & Parabens Propylene Glycol Carbomer (Carbopol 981) NaOH (10%) Scleroglucan Micropigment from Example 2 Titanium Dioxide	3.00 63.12 0.80 3.00 0.20 0.33 1.00 3.00 3.00

Example 28: O/W Emulsion

	<u>%</u>
Methyl Glucose Sequistearate	2.50
Tricontanyl PVP	1.00
Tocopherol & Ascorbyl Palmitate & Ascorbic Acid & Citric Acid & PEG-8	0.05
Decyl Oleate	4.00
Isopropyl Palmitate	6.00
Caprylic/Capric Triglyceride	5.00
Glyceryl Stearate Cetearyl Alcohol	1.00 1.00
2-[(2,4-Methoxy)phenyl]-4,6-bis[(2-hydroxy-4-methoxy)phenyl]-	2.00
(1,3,5)triazine	
Octyl Methoxycinnamate	5.00
Deionized Water	63.12
Phenoxyethanol & Parabens	0.80
Carbomer (Carbopol 981)	0.20
Glycerin NaOH (10%)	3.00 0.33
Scleroglucan	1.00
Micropigment from Example 1	4.00
Example 29: Lipcare composition	
Example 29. Lipcare composition	
	<u>%</u>
Glycerin	10.00
PEG-45 & Dodecyl Glycerol Copolymer	1.50
Quaternium-18 Bentonite Microcrystalline Wax	2.00 2.00
Beeswax	2.00
Glyceryl Stearate SE	53.00
Pentaerythrithil Stearate & Caprate & Caprylate Adipate	4.00
Castor Oil	4.00
Methylene Bis-benzotriazolyl Tetramethylbutylphenol Micropigment from Example 2	5.00
Titanium Dioxide	5.00 5.00
Zinc Oxide	5.00
Octyl Methoxycinnamate	4.00
Eucerinum anhydricum	ad 100

Example 30: W/O Emulsion

	,	<u>%</u>
PEG	i-30 Dipolyhydroxystearate	2.00
	tearyl Alcohol	20.00
Isos	tearic Acid	10.00
Octy	l Triazone	3.00
	nized Water	58.75
Glyc		5.00
	nylparaben	0.17
	ylparaben	0.03
MgS	O ₄ x7H₂O	0.75
Micro	opigment from Example 2	4.00
Exam	ple 31: O/W Emulsion	•
		<u>%</u>
	Dahashaand O Mathadalaana Bistoria	-
Α	Polyglyceryl-3 Methylglucose Distearate	2.0
	Decyl Oleate	5.7
	Isopropyl Palmitate	5.0
	Caprylic/Capric Triglyceride	6.5
	Octyl Methoxycinnamate	5.0
В	Glycerol	3.0
	Phenonip	0.5
	Deion. Water	62.9
С	Carbomer 141	0.2
	Isopropyl Palmitate	8.0
D	50% suspension from Example 8	8.0
_	•	
E	NaOH (10%)	as required

Example 32: O/W Emulsion

		<u>%</u>
Α	Polyglyceryl-3 Methylglucose Distearate Decyl Oleate Isopropyl Palmitate Caprylic/Capric Triglyceride	2.0 5.7 5.0 6.5
В	Glycerol Phenonip Deioniz. Water	3.0 0.5 62.9
С	Carbomer 141 Isopropyl Palmitate	0.2 0.8
D	Suspension from Example 2	6.0
E	NaOH (10%)	as required
Example 33: (O/W Emulsion)		
		<u>%</u>
Α	Polyglyceryl-3 Methylglucose Distearate	2.0
	Decyl Oleate	5.7
	Isopropyl Palmitate Caprylic/Capric Triglyceride	5.0
	Octyl Triazone	6.5 2.0
В	Glycerol	3.0
	Phenonip	0.5
	Water	62.3
С	Carbomer 141	0.2
	Isopropyl Palmitate	0.8
D	2,2'-Methylenebis(6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)phenol Micropigment Suspension (50%)	8.0
	Octyl Triazone Micropigment Suspension (50%)	4.0
Ε	NaOH (10%)	as required

Example 34: O/W Emulsion

		<u>%</u>		
Α	Polyglyceryl-3 Methylglucose Distearate	2.0		
	Decyl Oleate	5.7		
	Isopropyl Palmitate	5.0		
	Octyl Triazone	2.0 6.5		
	Caprylic/Capric Triglyceride	0.3		
В	Glycerol	3.0		
	Phenonip	0.5		
	Water	68.3		
С	Carbomer 141	0.2		
	Isopropyl Palmitate	8.0		
D	Micropigment from Example 2	6.0		
	Wild Opigine it from Example 2	0.0		
E	NaOH (10%)	as required		
Exan	Example 35: W/O Emulsion			
		<u>%</u>		
PF	6-30 Dipolyhydroxystearate (Arlacel P 135®)	3.00		
PEG-22/ Dodecyl Glycol Copolymer (Elfacos ST 37®)		1.00		
	ocrystalline Wax	1.00		
	rogenated Castor Oil	0.50		
	nesium Stearate	1.00		
	vi Stearate	15.00		
Coco Glycerides		2.00		
Mineral Oil		3.00		
Phenoxyethanol&Parabens		1.00 5.00		
Octyl Methoxycinnamate		0.10		
Dimethicone Water		54.40		
	nesium Sulfate (MgSO₄ x 7 H₂O)	1.00		
	pylene Glycol	4.00		
	Suspension from Example 3	8.00		

Example 36: W/O Emulsion

	<u>%</u>
Methoxy PEG-22/Dodecyl Glycol Copolymer (Elfacos E 200 [®])	3.00
PEG-22/Dodecyl Glycol Copolymer (Elfacos ST 37®)	3.00
Hydroxyoctacosanyl Hydroxystearate (Elfacos C 26®)	3.00
Octyl Stearate	15.00
Coco Glycerides	2.00
Mineral Oil	3.00
Phenoxyethanol & Parabens	1.00
4-Methylbenzylidene Camphor	3.00
Dioctyl Butamido Triazone	3.00
Dimethicone	0.20
Water	53.00
Phenylbenzimidazolesulfonic acid	3.00
Magnesium Sulfate (MgSO ₄ x 7 H₂O)	0.80
Propylene Glycol	4.00
Micropigment from Example 5	3.00
Wild opignion from Example o	0.00
Example 37: W/O Emulsion	
	<u>%</u>
Polyglyceryl-2 Dipolyhydroxystearate (Dehymuls PGPH [®])	2.00
PEG-30 Dipolyhydroxystearate (Arlacel P 135®)	2.00
Hydroxyoctacosanyl Hydroxystearate (Elfacos C 26 [®])	2.00
Zinc Stearate	1.00
Octyl Stearate	15.00
Coco Glycerides	2.00
Mineral Oil	3.00
Phenoxyethanol & Parabens	1.00
2,4-Bis{[4-(2-Ethylhexyloxy)-2-hydroxy]phenyl}-6-(4-	2.00
methoxyphenyl)1,3,5)triazine	2.00
Octyl Salicylate	3.00
Dimethicone	0,20
Water	56.70
Magnesium Sulfate (MgSO₄ x 7 H₂O)	1.00
Propylene Glycol	4.00
Micropigment from Example 6	5.00
	5.00

Example 38: W/O Emulsion

	<u>%</u>
Polyglyceryl-2 Dipolyhydroxystearate (Dehymuls PGPH®)	3.00
Glyceryl Oleate (Monomuls 90-O 18®)	1.00
Caprylic/Capric Triglyceride	6.00
Octyldodecanol	6.00
Cetearyl Isononaoate	5.00
Tocopheryl Acetate	1.00
Cera alba	1.20
Glycerin (86%)	5.00
Phenonip	0.50
Octyl Methoxycinnamate	4.00
Octyl Triazone	3.00
Micropigment from Example 3	5.00
Water	ad 100

Example 39: W/O Emulsion

	<u>%</u>
Polyglyceryl-2 Dipolyhydroxystearate (Dehymuls PGPH®)	3.00
Glyceryl Oleate (Monomuls 90-O 18®)	1.00
Caprylic/Capric Triglyceride	6.00
Octyldodecanol	6.00
Cetearyl Isononaoate	5.00
Octyl Methoxycinnamate	3.00
Tocopheryl Acetate	1.00
Cera alba	1.20
Glycerin (86%)	5.00
Phenonip	0.50
Micropigment from Example 10	5.00
Water	ad 100

Example 40: O/W Emulsion

Example 40: Of W Emulsion	
	<u>%</u>
Tego Care CG 90 (Goldschmidt AG)	6.00
Cetearyl Alcohol	1.50
Glycerylstearate	0.50
Octyldecanol	7.00
Capric/Caprylic Triglyceride	5.00
Cetearyl isononanoate	6.00
Octyl Methoxycinnamate	3.00
Deionized Water	51.14
Carbomer	0.20
NaOH (45%)	1.13
Glycerin	5.00
Methylparaben	0.17
Propylparaben	0.03
Terephthalylidenedibornanesulfonic acid	1.50
Micropigment from Example 5 (50% Suspension)	12.00
Example 41: O/W Microemulsion	·
	<u>%</u>
Ceteareth-12	8.0
Cetearyl Alcohol	4.0
Cetearyl isononanoate	20.0
Butyl Methoxydibenzoylmethane	2.0
Deionized Water	ad 100.0
Carbomer	0.2
Preservative	as required
Magnesium Sulfate (MgSO ₄ x 7 H₂O)	3.0
Micropigment from Example 9 (50% Suspension)	8.0
Example 42: O/W/O Emulsion	
	<u>%</u>
Polyglyceryl-2 polyhydroxystearate	5.0
Mineral oil	12.5
Stearic acid	2.0
Cetearyl isononanoate	12.5
Methylbenzylidene Camphor	2.0
Homosalate	2.0
Deionized Water	ad 100.0
Carbomer	0.2
Preservative	as required
NaOH	as required

Micropigment from Example 2 (50% Suspension)

Example 43: O/W Emulsion

·	<u>%</u>
Glycerin Stearate/Polyethylene glycol(MW100) stearate	3.0
Cetyl/Stearyl Alcohol 20EO (Eumulgin B 2)	1.0
Cetyl/Stearyl Alcohol (Lanette O)	2.0
Caprylic/Capric triglyceride (Myritol 318)	4.0
Dicaprylyl ether	6.0
Mineral oil and Quaternium-18 Hectorite	3.0
Glycerin stearate, Cetyl/stearyl Alcohol, Cetyl palmitate, coco	2.0
glycerides (Cutina CBS)	
4-Methylbenzylidene Camphor	1.0
Octyl Triazone	2.0
Deionized Water	ad 100.0
Glycerin, 85%	3.0
Preservative	as required
Magnesium aluminium silicate (Vegum Ultra)	0,3
NaOH	as required
Micropigment from Example 2 (50% Suspension)	10.0

Example 44:

Into the suncare product "Sensitive Skin" (children) from Lancaster (Monaco), characterized by the following ingredients: TiO₂, ZnO and Aqua, Didecene, Glycerine, Cyclomethicone, Shea Butter, Sweet Almond Oil, Polyglycerin-4, Urea, Aluminium Starch, Octenyl succinate, Alumina, Parfum, MgSO₄, Silica, NaCl, Tocopheryl acetate, Caffeine, PVP/Eicosene Copolymer, Shellac, Simethicone, Phenoxyethanol, NaLactate, Methylsilanol, Menthyl Lactate, Allantoin, Bisabolol, Glycine, Panthenol, Propylene Glycol, Stoneroot Extract, Lecithin, Algae Extract, Methyldibromo Glutaronitrile, PVP, Citric Acid, Copper Gluconate, Ascorbic Acid, Ascorbyl Palmitate, PEG-8, Tocopherol, Acerola, Aloe Barbadensis Gel, Melanin, Alcohol denat. Dimethicone, Guar Hydroxypropyltrimonium Chloride, Dextrin, Glycoproteins Iron oxides, were subsequently mixed 4% of micronized 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)phenol (d50 = 200 nm). The original SPF of 15 increased as a result to 25 and, following storage for a few days, increased again to an SPF of 31.

Example 45:

Into the sun milk "Active Sun Care Sensitive Skin" from Marbert Cosmetics, Düsseldorf, characterized by the following ingredients: TiO₂, Benzophenone-3, Isoamyl p-Methoxycinnamate, and Aqua, C₁₂₋₁₅ Alkyl benzoate, Caprylic/Capric Triglyceride, Cyclomethicone, Glycerine, Glyceryl Stearate, Cetearyl Alcohol, Tocopheryl acetate, Stearic Acid, Palmitic Acid, Parfum, NaCocoyl Lactylate, Xanthan Gum, Bisabolol, DMDM

Hydantoin, PVM/MA Decadiene Crosspolymer, Polyhydroxystearic acid, Alumina, NaOH, Glucose, Iodopropynyl Butylcarbamate, Carrageenan, Silica and Glucuronic acid, were subsequently mixed 4% of micronized 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)phenol (d50 = 200 nm). The original SPF of 6 increased as a result to 13 and, after storage for a few days, increased again to an SPF of 16.

Example 46:

Into the sunscreen emulsion "Delial Sonnenmilch 10" from Sara Lee, Düsseldorf, characterized by the following ingredients: Octyl Methoxycinnamate, NaPhenylbenzimidazole Sulfonate, Butyl Methoxy Dibenzoylmethane and Aqua, Paraffinum liquidum, Alcohol denat., Isopropyl Palmitate, Glycerine, Cetearyl Alcohol, Glyceryl Stearate SE, Tocopheryl acetate, Phytantriol, Ascorbyl Palmitate, PEG-40 Castor Oil, NaCetearyl Sulfate, Dimethicone, Na-Carbomer, Na₂-EDTA and Parfum, were subsequently mixed 4% of micronized 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)phenol (d50 = 200 nm). The original SPF of 10 increased as a result to 18 and, after storage for a few days, increased again to an SPF of 28.

Example 47:

Into the sun protection formulation "Ambre Solaire" SPF 12 from Laboratoires Garnier, Paris/Karlsruhe, characterized by the following ingredients: TiO₂, Octocrylene, Butyl Methoxy Dibenzoylmethane, Terephthalylidene dicamphor sulfonic acid and Aqua, Cyclopentasiloxane, Glycerine, Propylene glycol, Isohexadecane, Stearic acid, Octyl palmitate, Stearyl heptanoate, PVP/Eicosene Copolymer, K-Cetyl Phosphate, Buxus chinensis, Tocopheryl acetate, Hydroxypropyl Methylcellulose, Phenoxyethanol, Stearyl caprylate, PEG-100 Stearate, Ethylparaben, Triethanolamine, Dimethiconol, Dimethicone, Propylparaben, Acrylates/C₁₀₋₃₀-Alkyl acrylate crosspolymer, Na₂-EDTA, Butyrospermum parkii, Cetyl Alcohol, Methylparaben, Butylparaben, BHT, Aluminium hydroxide, Glyceryl Stearate were subsequently mixed 4% of micronized 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)phenol (d50 = 200 nm). The original SPF of 12 increased as a result to 18 and, after storage for a few days, increased again to an SPF of 28.

Example 48:

Into the sunscreen formulation "Ambre Solaire" SPF 6 from Laboratoires Garnier, Paris/Karlsruhe, characterized by the following ingredients: TiO₂, Octocrylene, Butyl

Methoxy Dibenzoylmethane, Terephthalylidene dicamphor sulfonic acid and Aqua,

Cyclomethicone, Glycerine, Propylene glycol, Isohexadecane, Stearic acid, Octyl palmitate, Stearyl heptanoate, PVP/Eicosene Copolymer, K-Cetyl Phosphate, Buxus chinensis, Tocopheryl acetate, Hydroxypropyl Methylcellulose, Phenoxyethanol, Stearyl caprylate, PEG-100 Stearate, Ethylparaben, Triethanolamine, Dimethiconol, Dimethicone, Propylparaben, Acrylates/C₁₀₋₃₀-Alkyl acrylate crosspolymer, Na₂-EDTA, Butyrospermum parkii, Cetyl alcohol, Methylparaben, Butylparaben, BHT, Aluminium hydroxide, Glyceryl stearate and Parfum, were subsequently mixed 4% of micronized 2,2'-methylenebis[6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)phenol (d50 = 200 nm). The original SPF of 6 increased as a result to 16 and, after storage for a few days, increased again to an SPF of 21.

Example 49: Prevention of the increase in skin tanning by a micronized UV absorber Methylene Bis-benzotriazolyl Tetramethylbutylphenol

Method:

20 volunteers of direct Asian origin (father and mother) who have not been directly exposed to the sun for the past 3 months, to whom an explanation of the study has been given, from whom a declaration of consent has been obtained and who have satisfied the inclusion conditions, are treated twice daily on the test sites on the upper thigh for three weeks with a cream containing 2,2'-methylenebis(6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)-phenol or with a placebo cream.

The volunteers are irradiated on the test sites on the upper thigh 3x weekly with 0.2 to 05 MED UVAB.

The first application of the preparations takes place after the first irradiation. Evaluation and irradiation are carried out after each application of the test products. Comparable untreated irradiated, or untreated nonirradiated areas serve as reference.

The colour values of the test fields are documented in each case using a Minolta CM-508i camera as L*a*b* values in accordance with DIN 5033, ISO 7724/1, JIS Z8722.

The colour and lightness changes are determined for each subject and ascertained as the difference between the respective skin colour of the untreated, nonirradiated reference area and the test areas. These values are averaged over all subjects and given as L*, a* and b* values.

Test preparations:

(A): Composition comprising 6% of 2,2'-Methylenebis(6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)phenol), Water, Octyl stearate, Coco glycerides, Propylene glycol, Methoxy-PEG-22/Dodecyl glycol copolymer, PEG-22/Dodecyl glycol copolymer, Hydroxyoctacosanyl hydroxystearate, Mineral oil, Phenoxyethanol & Parabens, Magnesium sulfate heptahydrate, Dimethicone, Allantoin.

(B): Composition comprising 3% of 2,2'-Methylenebis(6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)phenol), Water, Octyl stearate, Coco glycerides, Propylene glycol, Methoxy-PEG-22/Dodecyl glycol copolymer, PEG-22/Dodecyl glycol copolymer, Hydroxyoctacosanyl hydroxystearate, Mineral oil, Phenoxyethanol & Parabens, Magnesium sulfate heptahydrate, Dimethicone, Allantoin.

(C): Placebo comprising Water, Octyl stearate, Coco glycerides, Propylene glycol, Methoxy-PEG-22/Dodecyl glycol copolymer, PEG-22/Dodecyl glycol copolymer, Hydroxyoctacosanyl hydroxystearate, Mineral oil, Phenoxyethanol & Parabens, Magnesium sulfate heptahydrate, Dimethicone, Allantoin.

L*a*b* values compared with nonirradiated skin following repeated UVAB irradiation (3 x weekly) and in the case of the application of compositions (A) and (B).

<u>Preparation</u>	Lightness L*			Red	Red component a*			Yellow component b*		
Number of irradiations	3	6	9	3	6	9	3	6	9	
Placebo	-5.32	-12.01	-14.01	4.90	2.33	0.52	3.09	6.82	7.93	
(B)	-1.05	-5.23	-7.13	0.45	0.77	-0.03	1.11	2.63	3.49	
(A)	2.18	8.26	11.40	0.24	0.45	0.39	-0.38	-0.69	-0.21	
Irradiated untreated	-5.19	-12.38	-14.55	5.13	1.77	-0.19	2.64	6.39	7.44	

Discussion of the results:

Lightness

While the placebo-treated and the untreated irradiated areas decrease in lightness to roughly the same extent, i.e. become darker, this effect is considerably less in the case of

the application of the composition (B) over the time. In the case of the application of composition (A), lightening of the skin is found.

Reddening

The red component of the irradiated skin is most intense after 3 irradiations and drops back to the normal value by the end of the irradiations. The increase in the red component corresponds to the development of a UV-induced erythema, which arises only to a low degree in the case of the application of compositions (A) or (B).

Yellow component

The yellow component increases both in the case of the application of placebo and in the untreated irradiated control area. The increase is much less in the case of the application of composition (B) and is prevented in the case of the application of composition (A).

What is claimed is:

- 1. A method for preventing tanning and for lightening human skin and hair which comprises applying to the hair and skin micronized organic UV filters.
- 2. A method according to claim 1, wherein the organic UV filters are chosen from triazine or benzotriazole derivatives, amides containing a vinyl group, cinnamic acid derivatives, sulfonated benzimidazoles, Fischer base derivatives, diphenylmalonitriles, oxalylamides, camphor derivatives, diphenylacrylates, paraaminobenzoic acid (PABA) and derivatives thereof, salicylates and benzophenones.
- 3. A method according to claim 1, wherein the organic UV filters are chosen from triazine derivatives of the formula

in which

- R_1 , R_2 and R_3 , independently of one another, are hydrogen; OH; C_1 - C_{18} alkoxy; -NH₂; -NH-R₄; -N(R₄)₂; -OR₄,
- R₄ is C₁-C₅alkyl; phenyl; phenoxy; anilino; pyrrolo, wherein phenyl, phenoxy, anilino or pyrrolo may be unsubstituted or substituted by one, two or three OH groups, carboxyl, -CO-NH₂, C₁-C₅alkyl or C₁-C₅alkoxy; a methylidenecamphor group; a group of the formula -(CH=CH)_mC(=O)-OR₄; a group of the formula

di- or tri-C1-C4alkylammonium, mono-, di- or tri-C2-C4alkanolammonium salts, or

$$C_1$$
- C_3 alkyl esters thereof; or a radical of the formula (1a) $-(CH_2)_{m_1} \cap C_{m_2} \cap C_{m_3}$;

R₅ is hydrogen; unsubstituted C₁-C₅alkyl or C₁-C₅alkyl substituted by one or more OH groups; C₁-C₅alkoxy; amino; mono- or di-C₁-C₅alkylamino; M; a radical of the formula

(1e)
$$-N \longrightarrow_{CO_2R_6}$$
; in which

R', R" and R"', independently of one another, are unsubstituted C₁-C₁₄alkyl or C₁-C₁₄alkyl substituted by one or more OH groups;

 R_6 is hydrogen; M; C_1 - C_5 alkyl; or a radical of the formula -(CH_2)_{m_2}-O- T_1 ;

M is a metal cation;

T₁ is hydrogen; or C₁-C₈alkyl;

m is 0 or 1

m₂ is 1 to 4; and

m₃ is 2 to 14.

4. A method according to claim 1, wherein the organic UV filters are chosen from triazine derivatives of the formula

in which

R₇ and R₈, independently of one another, are C₁-C₁₈alkyl; C₂-C₁₈alkenyl; a radical of the formula -CH₂-CH(-OH)-CH₂-O-T₁; or

 R_7 and R_8 are a radical of the formula (2a) $R_9 = \begin{bmatrix} R_{10} \\ \vdots \\ R_{11} \end{bmatrix} \begin{bmatrix} R_{10} \\ \vdots \\ R_{11} \end{bmatrix}$

 R_9 is the direct bond; a straight-chain or branched C_1 - C_4 alkylene radical or a radical of the formula $-c_{m_1}H_{\frac{1}{2m_1}}O_-$;

R_{10} , R_{11} and R_{12} , independently of one another, are C_1 - C_{18} alkyl; C_1 - C_{18} alkoxy or a radical of

the formula
$$-0.8i - R_{13}$$
; R_{13} ; R_{13} ;

 R_{13} is C_1 - C_5 alkyl;

m₁ is 1 to 4;

p₁ is 0 to 5;

A₁ is a radical of the formula

 R_{14} is hydrogen; C_1 - C_{10} alkyl, -(CH_2CHR_{16} -O) $_{n_1}$ - R_{15} ; or a radical of the formula

-CH₂-CH(-OH)-CH₂-O-T₁;

 R_{15} is hydrogen; M; C_1 - C_5 alkyl; or a radical of the formula $-(CH_2)_{m_2}$ - $O-(CH_2)_{m_3}$ - T_1 ;

R₁₆ is hydrogen; or methyl;

T₁ is hydrogen; or C₁-C₈alkyl;

Q₁ is C₁-C₁₈alkyl;

M is a metal cation;

 m_2 and m_3 , independently of one another, are 1 to 4; and

n₁ is 1 to 16.

5. A method according to claim 1, wherein the organic UV filters are chosen from triazine derivatives of the formula

(3)
$$R_{23}$$
 R_{22} R_{22} R_{24} R_{24} R_{24}

in which

R₂₁ is C₁-C₃₀alkyl; C₂-C₃₀alkenyl; unsubstituted C₅-C₁₂cycloalkyl or C₅-C₁₂cycloalkyl monoor polysubstituted by C₁-C₅alkyl; C₁-C₅alkoxy-C₁-C₁₂alkyl; amino-C₁-C₁₂alkyl; C₁-C₅monoalkylamino-C₁-C₁₂alkyl; C₁-C₅dialkylamino-C₁-C₁₂alkyl; a radical of the

formula (3a)
$$-(CH_2)\frac{1}{n_1}(O)\frac{1}{m_1}$$
 ; or (3b) ; in which

 R_{22} , R_{23} and R_{24} , independently of one another, are hydrogen, -OH; C_1 - C_{30} alkyl, C_2 - C_{30} alkenyl,

R₂₅ is hydrogen; or C₁-C₅alkyl;

m, is 0 or 1; and

n₁ is 1 to 5.

6. A method according to claim 1, wherein the organic UV filters are chosen from triazine derivatives of the formula

(4)
$$R_{26}$$
 is N_{CH_2} ; in which R_{26} is N_{CH_3} ; and

r and s, independently of one another, are 0 to 20.

7. A method according to claim 1, wherein the organic UV filters are chosen from triazine derivatives of the formula

8. A method according to claim 1, wherein the organic UV filters are chosen from triazine derivatives of the formula

9. A method according to claim 1, wherein the organic UV filters are chosen from triazine derivatives of the formula

10. A method according to claim 1, wherein the organic UV filters are chosen from triazine derivatives of the formula

 R_{27} , R_{28} and R_{29} , independently of one another, are a radical of the formula

(25c)
$$R_{31}$$
 R_{32} O OR_{30}

R₃₀ is hydrogen; alkali metal; an ammonium group -N(R₃₃)₄,

R₃₃ is hydrogen; C₁-C₅alkyl; or a polyoxyethylene radical which has 1 to 10 ethylene oxide units and the terminal OH group can be etherified with a C₁-C₅alcohol;

R₃₁ is hydrogen; -OH; or C₁-C₆alkoxy;

R₃₂ is hydrogen or -COOR₃₀; and

n is 0 or 1.

11. A method according to claim 1, wherein the organic UV filters are chosen from benzotriazole derivatives of the formula

(26)
$$N$$
 N N , in which

T₁ is C₁-C₅alkyl or hydrogen; and

T₂ is C₁-C₅alkyl or phenyl-substituted C₁-C₅alkyl.

12. A method according to claim 1, wherein the organic UV filters are chosen from benzotriazole derivatives of the formula

T₂ is C₁-C₄alkyl or phenyl-substituted C₁-C₅alkyl.

13. A method according to claim 1, wherein the organic UV filters are chosen from Fischer base aldehydes of the formula

(32)
$$R_{41}$$
 R_{42} R_{44} , in which R_{43}

R₄₁ is hydrogen; C₁-C₅alkyl; C₁-C₁₈alkoxy; or halogen;

 R_{42} is C_1 - C_8 alkyl; C_5 - C_7 cycloalkyl; or C_6 - C_{10} aryl;

 R_{44} is hydrogen; or a radical of the formula $-C_{00}$

$$R_{45}$$
 is $\begin{bmatrix} R_{47} \\ N \end{bmatrix}_{n}^{R_{46}} C = 0$; C_1 - C_{18} alkoxy; or a radical of the formula

 R_{46} and R_{47} , independently of one another, are hydrogen; or C_1 - C_5 alkyl; R_{48} is hydrogen; C_1 - C_5 alkyl; C_5 - C_7 cycloalkyl; phenyl; phenyl- C_1 - C_3 alkyl; R_{49} is C_1 - C_{18} alkyl;

n is 0 or 1.

14. A method according to claim 1, wherein the organic UV filters are chosen from compounds of the formula

(33)
$$ZO_3S$$

$$R_{54}$$

$$C_m - C_n R_{53}$$

$$R_{54}$$

$$R_{53}$$

$$R_{54}$$

$$R_{53}$$

$$R_{54}$$

$$R_{53}$$

$$R_{54}$$

$$R_{53}$$

$$R_{54}$$

$$R_{53}$$

$$R_{54}$$

$$R_{54}$$

$$R_{55}$$

$$R_{51}$$

$$R_{51}$$

in which

R₅₀, R₅₁, R₅₂, R₅₃, R₅₄, independently of one another, are hydrogen, C₁-C₈alkyl or C₅-C₁₀cycloalkyl;

R₅₅ is hydrogen; C₁-C₈alkyl; C₅-C₁₀cycloalkyl; hydroxyl; C₁-C₈-alkoxy; COOR₅₆; or CONR₅₇R₅₈;

 R_{56} , R_{57} and R_{58} , independently of one another, are hydrogen or C_1 - C_6 alkyl;

X and Y, independently of one another, are hydrogen, -CN; CO₂R₅₉; CONR₅₉R₆₀; or COR₅₉; where the radicals X and Y may additionally be a C₁-C₈alkyl radical, a C₅-C₁₀alkyl radical or a heteroaryl radical having 5 to 6 ring atoms, where, in addition, X and Y or

- R₅₀ together with one of the radicals X and Y can represent the radical to complete a 5- to 7-membered ring which may contain up to 3 heteroatoms, where the ring atoms may be substituted by exocyclically double-bonded oxygen and/or C₁-C₈alkyl and/or C₅-C₁₀cycloalkyl radicals, and/or may contain C=C double bonds;
- Z is hydrogen; ammonium; alkali metal ion; or the cation of an organic nitrogen base used to neutralize the free acid group;

R₅₉ and R₆₀, independently of one another, are hydrogen, C₁-C₈alkyl or C₅-C₁₀cycloalkyl; and

n and m, independently of one another, are 0 or 1.

- 15. A method according to claim 1, wherein the organic UV filters are used as mixtures.
- 16. A process for the preparation of mixtures of the organic UV filters defined in claim 1 which can be used according to the invention, which comprises thoroughly mixing the UV filters present in micronized form together.

- 17. A process for the preparation of mixtures of the organic UV filters defined in claim 1 and which can be used according to the invention, which comprises micronizing the organic UV filters as mixtures of at least two individual substances.
- 18. A process for the preparation of mixtures of the organic UV filters defined in claim 1 and which can be used according to the invention, which comprises melting together at least two individual substances, cooling the melt, and then subjecting the resulting composite to a micronization process.
- 19. A composite obtainable by melting together at least two of the organic UV filters defined in claim 1.
- 20. A method according to claim 1, wherein an inorganic pigment is additionally mixed in.
- 21. A method according to claim 20, wherein the inorganic pigments are chosen from TiO₂, ZnO, iron oxides, mica and Ti or zinc salts of organic acids.
- 22. A composite obtainable by melting together at least two of the organic UV filters defined in claim 1 and at least one of the inorganic pigments defined in claim 20.
- 23. A method according to claim 1, wherein an antioxidant is additionally mixed in.
- 24. A method according to claim 23, wherein the antioxidant is chosen from tocopherols, ellagic acid, propyl gallate, butylated hydroxytoluene, butylated hydroxyanisole, 2,4,6-tris(3,5-di-t-butyl-4-hydroxybenzyl)mesitylene, tetrakis[methylene-3-(3',5'-di-t-butyl-4'-hydroxyphenyl)propionate]methane, the compound of the formula

acid, rutic acid derivatives; urocanic acid, urocanic acid derivatives and propolis.

- 25. A composite obtainable by melting together at least two of the organic UV filters defined in claim 1 and at least one of the antioxidants defined in claim 23, and, if desired, one or more inorganic pigments.
- 26. A method according to claim 1, wherein a cationic or anionic compound is mixed in.
- 27. A method according to claim 26, wherein the cationic or anionic compound is chosen from camphorbenzalkonium methosulfates, fatty amines, betaines, quats, citric monoglyceride, sodium methylcocoyltaurate, phospholipids, ceramides and phytosterols.
- 28. A composite obtainable by melting together at least two of the organic UV filters defined in claim 1 and at least one of the cationic or anionic compounds defined in claim 26.
- 29. A method according to claim 1, wherein a pharmaceutical or cosmetic active ingredient is additionally mixed in.
- 30. A cosmetic formulation comprising one of the organic UV filters defined in claim 1, if desired one or more antioxidants and/or inorganic pigments and/or a cationic or anionic compound, and cosmetically compatible carriers or auxiliaries.

- 31. A cosmetic formulation according to claim 30, which additionally comprises an oil-soluble, nonmicronized UV filter.
- 32. A pharmaceutical formulation comprising a mixture of at least two of the organic UV filters defined in any one of claims 1 to 15, if desired one or more antioxidants and/or inorganic pigments and/or a cationic or anionic compound, and pharmaceutically compatible carriers or auxiliaries.

Abstract

The invention describes the use of micronized organic UV filters for preventing tanning and for lightening human skin and hair, and their use in cosmetic and pharmaceutical formulations.

The micronized UV filters used according to the invention cover a broad UV spectrum and therefore have excellent sunscreen properties.